U.S. DEPARTMENT OF COMMERCE
National Technical Information Service

PB-258 162

Destruction of Polychlorinated Biphenyls in Sewage Sludge During Incineration

Versar, Inc, Springfield, Va

Prepared for

Environmental Protection Agency, Washington, D C

1976

273084

PB 258 162

IDENTIFICATION OF POLYCHLORINATED BIPHENYLS IN [illegible]

FINAL REPORT

by

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Contract 68-01-1587

Prepared for

Environmental Protection Agency
Washington, D. C.

[illegible]
NATIONAL TECHNICAL
INFORMATION SERVICE
[illegible]

BIBLIOGRAPHIC DATA SHEET	2. Report No.	3. Recipient's Accession No.
4. Title and Subtitle: Destruction of Polychlorinated Biphenyls in Sewage Sludge During Incineration.		5. Report Date: 1978
7. Author(s): Frank C. Whitmore		8. Performing Organization Rept. No.
9. Performing Organization Name and Address: Versar, Inc., Springfield, Va.		10. Project/Task/Work Unit No.
		11. Contract/Grant No.: EPA-68-01-1587
12. Sponsoring Organization Name and Address: Environmental Protection Agency, Washington, D. C.		13. Type of Report & Period Covered
		14.

15. Supplementary Notes:

16. Abstract:

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17. Key Words and Document Analysis. 17a. Descriptors:

*Sludge disposal,
*Incinerators,
*Air pollution,
*Solid waste disposal,
Sewage sludge,
Chlorine aromatic compounds,
Decomposition,
Flue gases,
Gas sampling,
Gas analysis

17b. Identifiers/Open-Ended Terms:

*Biphenyl/chloro,
*Polychlorinated biphenyls.

17c. COSATI Field/Group: 13B

18. Availability Statement: National Technical Information Service, Springfield, Va. 22161	19. Security Class (This Report): UNCLASSIFIED	21. No. of Pages: 80p.
	20. Security Class (This Page): UNCLASSIFIED	22. Price: $5.00/3.00

ABSTRACT

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EXECUTIVE SUMMARY

The concern as to the significance of municipal sewage sludge incinerators as primary sources for the injection of polychlorinated biphenyls (PCB's) into the biota has resulted in the tentative imposition of a requirement of very high afterburner temperatures (870°C) coupled with long holdup times (of the order of 2 seconds) in order to fully combust these compounds. An experiment has been conducted to determine the stability of PCB's in such a municipal multiple hearth furnace operating at normal temperatures (afterburner temperature 635°C) and with normal holding times (less than 0.5 sec). The test material, Aroclor 1254, which is an admixture of some 16 to 20 PCB's, was injected into the sludge cake at a rate of 50 ppm based on the solids content of the sludge cake. After sufficient time had elapsed to allow equilibrium to be established (1 hour with dwell time of the order of 45 minutes), samples were taken of the afterburner gases, the stackgases, scrubber water output and ash (from the bottom hearth). This set of sampling data was repeated a second and a third time.

The results of the experiment demonstrate several facts of significance:

(a) There was no PCB found in either scrubber water effluent or in the ash.

(b) A relatively large percentage of the injected PCB was found to be volatilized as the sludge dried. The combustion apparently proceeded with great rapidity in the high temperature, water saturated gas stream prior to quenching.

(c) The net amount of volatile PCB's in the exhaust stream (stack) indicated that less than 6 per cent of the injected PCB's escaped destruction.

(d) Very little PCB was found on the particulate matter in either the afterburner bricks or in the stack exhaust; a fact pointing to the volatilization of the PCB.

These experiments suggest that satisfactory reductions in the level of injection of undesired PCB's into the atmosphere can be accomplished without the imposition of the rather extreme requirement of significantly increased afterburner temperature and marked lengthening of retention time of the hot gases.

1.0 INTRODUCTION

The polychlorinated biphenyls (PCB's) constitute a large class of relatively stable organic compounds derived from biphenyl which are widely used as heat transfer agents, industrial solvents, pesticides, and intermediates in a wide variety of organic syntheses. Because of the high resistance to natural degradation processes exhibited by these compounds, there has been an alarming increase, on a world-wide basis, in the background levels of the PCB's. Even more disturbing is that much of the background is found to be fixed in various portions of the biota, including humans. Since many of these compounds are toxic, at least at significant concentrations, there is much concern about the possible long term effects of chronic exposure to even background levels.

The increasing tendency to dispose of sewage sludge by incineration, coupled with the observed relatively high level of PCB's found in sludge (ranging up to 10 ppm), suggests a real possibility of a relatively massive recycling mechanism for the PCB's. A significant fraction of the refractory organics originally in the sludge could, if not contained, be ejected into the effluent air stream from whence they could be widely disseminated. It is therefore important to understand the conditions under which sewage sludge incineration can be accomplished with the assurance that such compounds as the PCB's contained therein are destroyed.

BACKGROUND AND EXPERIMENTAL APPROACH

In a recently completed study, Versar Inc. has been able to demonstrate that coincineration of such refractory compounds as HCP with sewage sludge in a practical process resulting in 99.9+ per cent destruction of the HCP. The close structural similarity between the PCB's and HCP suggests that the conditions existing within a multiple hearth sludge incinerator should be capable of a similar destruction of the PCB's.

In order to determine the validity of this assumption, an experiment has been conducted at the Palo Alto municipal incinerator in which the sludge input to the incinerator was deliberately doped with a suitable PCB preparation at a level of 50 ppm (dry weight). After allowing the furnace to come to equilibrium, assuming that the residence time was on the order of 45 minutes, triplicate samples were taken of the scrubber outflow, the ash, the hot gas stream at the high temperature branch prior to the scrubber and at the stack just before emergence into the outside air. In addition a series of background samples were taken of each stream including the sludge.

2.0 EXPERIMENTAL CONDITIONS

2.1 Furnace Operating Conditions

The operation of the furnace was normal during the course of the experiment as is indicated by the data in Table I (Table I is presented in English units since most municipal installations utilize control and monitoring equipment calibrated in English units - the corresponding metric unit data are presented in Table IA).

2.2 Feed System and Test PCB

Referring to Figure 1, which is a schematic representation of the multiple hearth furnace at Palo Alto, the feed, in the form of a solution in kerosene was fed from a burette into the sludge cake feed screw at a rate of 22.5 grams per hour. The test PCB, which was kindly furnished by Monsanto Chemical Co., was a preparation Aroclor 1254 which is a combination of some 14 to 16 PCBs. A gas chromatographic spectrum of the test PCB is shown as Figure 2.

2.3 Sampling Points

Referring to Figure 1, sampling of the gas stream was carried out at the exhaust port from the top hearth of the furnace (position labeled

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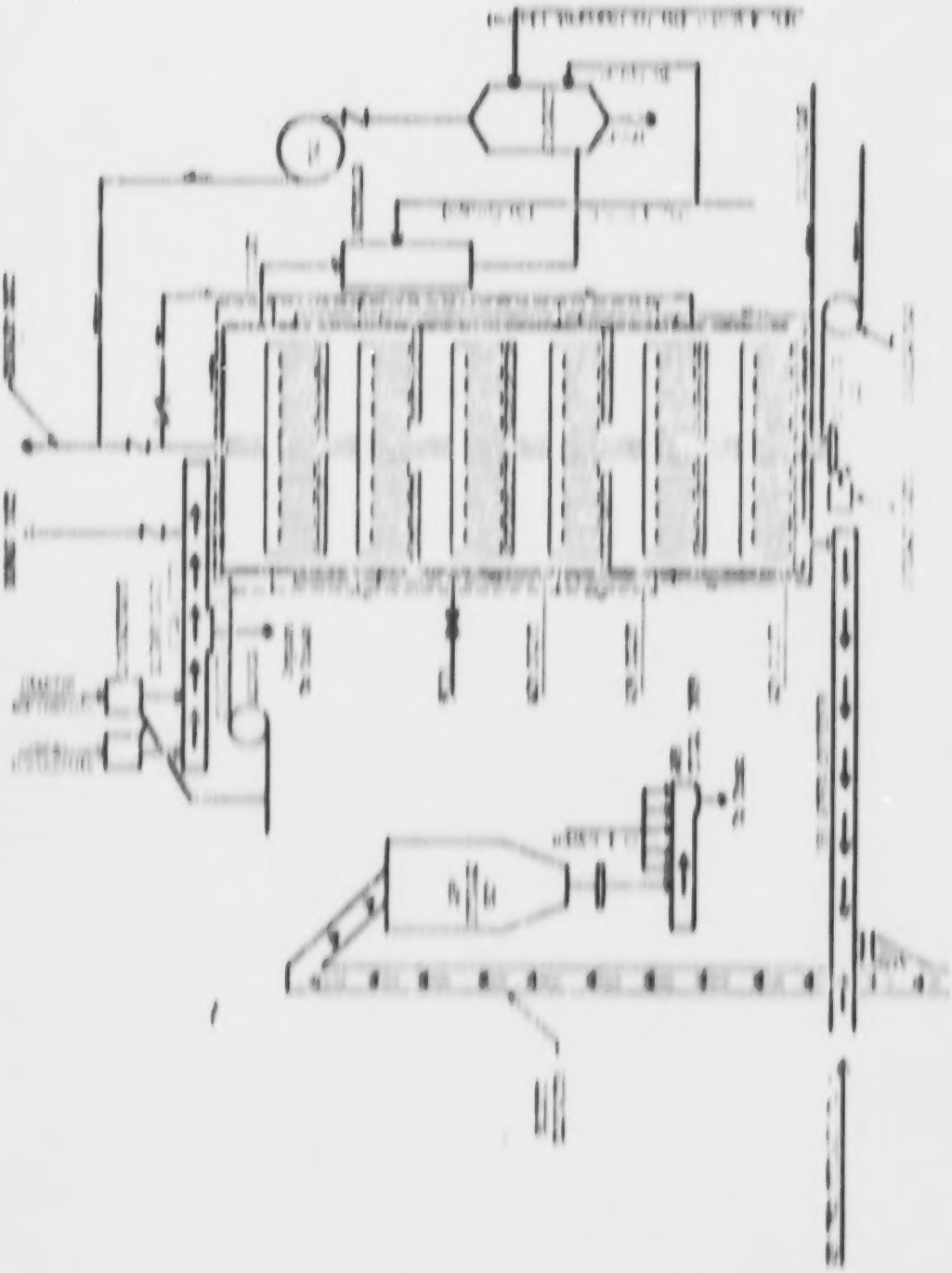


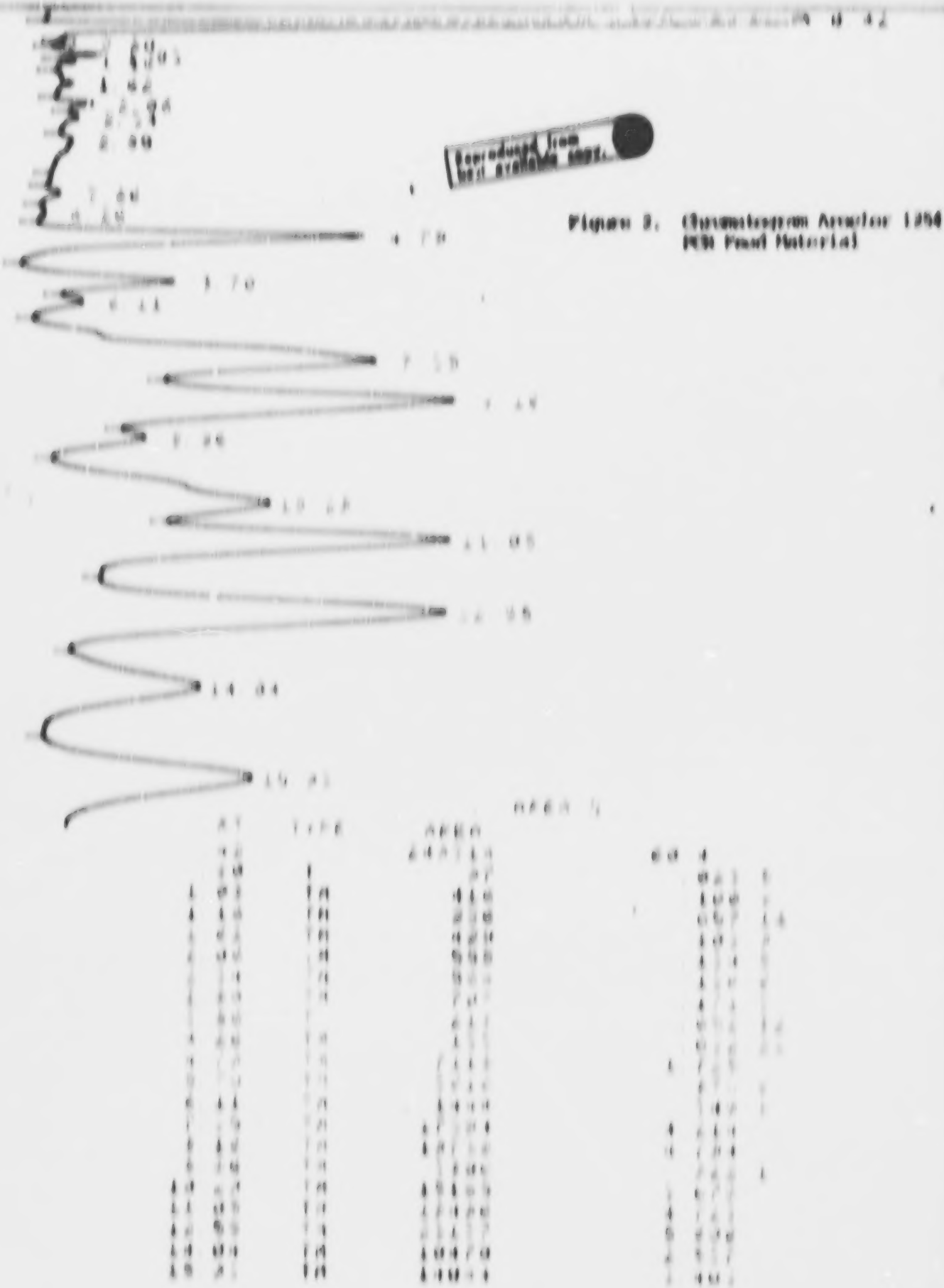


Figure 3. Chromatogram Acrylor 1254 PCB Fluid Material

exhaust) just upstream of the precooler and at the exhaust duct just downstream of the fan. Sampling was accomplished using EPA standard method for particulates in stationary sources with a 0.45 micron filter followed by an impinger train using 2,2,4 trimethyl pentane (iso-octane) as the collection fluid. In addition samples were taken of the ash from the sixth (bottom) fourth of the furnace and of the scrubber outflow at the scrubber drain.

3.4 Gas Sampling

Gas velocity measurements were taken during sampling in order to allow computation of flow rates of the sampled stream. The two pitot assemblies used were calibrated as shown in Table 2 where v^2 was obtained with a Dwyer standard pitot and the Dwyer calibration curves. From the C values reported in Table 2, velocities were subsequently computed using the relationship displayed in Table 2.

The geometry of the aftertunnel breaching showing the available sampling ports is shown in Figure 3. The geometry of the stack assembly and the location of the available sampling port is shown in Figure 4. In the case of the aftertunnel breach, geometric considerations made the use of the middle port mandatory (the layout of the aftertunnel breach, the precooler duct and the scrubber top assembly was such that the sampling tube and impinger train could not be inserted into the lower ports). The positions on the traverse for both aftertunnel breach and stack are displayed in Table 3 for reference.

A typical set of velocity pressure measurements is displayed in Table 4 along with the method by which the average velocity and hence the gas flow rate through the exhaust stack is calculated. Although the velocity distribution shown in Table 4 is not quite symmetrical by a laminar flow, no corrections were made for this.

TABLE 3

PITOT TUBE CALIBRATION TO DETERMINE C FACTOR

Sample Point	Δp IN. H_2O	$\sqrt{\Delta p}$	T_{avg} (°R)	V' (FT/Min.)	V^0 (FT/Min.)	C	C
Level 1/8" remain	.015	.123	1120	14.14	13.17	.93	.94
	.02	.142	1100	16.32	15.17	.93	
	.03	.171	1100	19.66	18.33	.93	
	.035	.188	1100	18.16	17.5	.96	
Mount 1/4" remain	.78	.883	800	66.2	65.3	.987	.99
	.74	.860	800	64.48	63.3	.982	
	.67	.818	800	61.33	60.67	.989	
	.59	.768	800	57.58	57.33	.996	
	.57	.755	800	56.61	56.17	.992	

The relationship between pitot pressure and velocity is given by

$$v(ft/min) = 85.4 K_p C \sqrt{\frac{T}{Mp}}$$

Where Δp = pitot pressure in in. H_2O

T = absolute temperature at sampling site
M = gaseous molecular weight
p = absolute pressure in in. Hg
C = calibration constant for probe

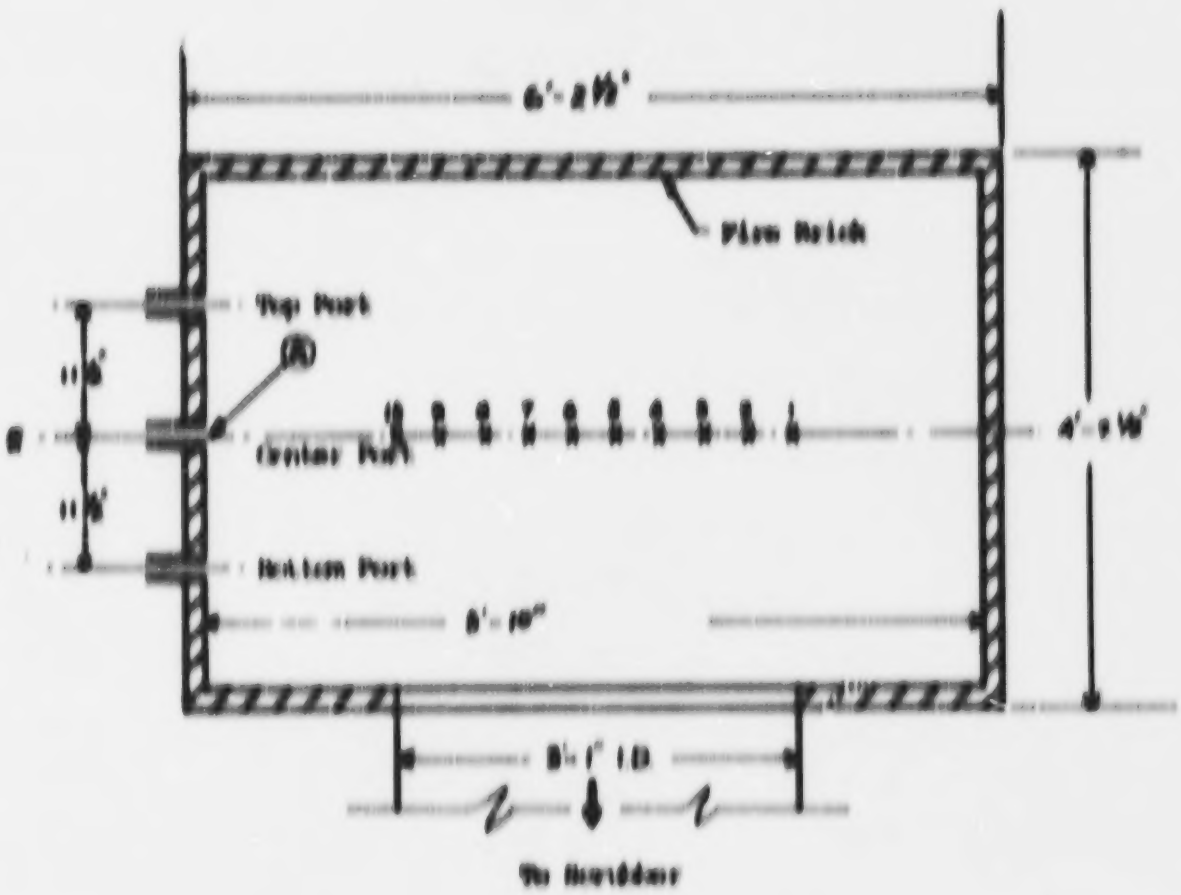


Figure 1. High Temperature Breach Showing Location of Sampling Ports and Traverse Points. (View looking into Furnace)

Positions for Sampling Points (Refer to Figure 1)

Position	Location Relative to Point (A)
1	54"
2	50"
3	46"
4	42"
5	38"
6	34"
7	30"
8	26"
9	22"
10	18"

Figure 2.

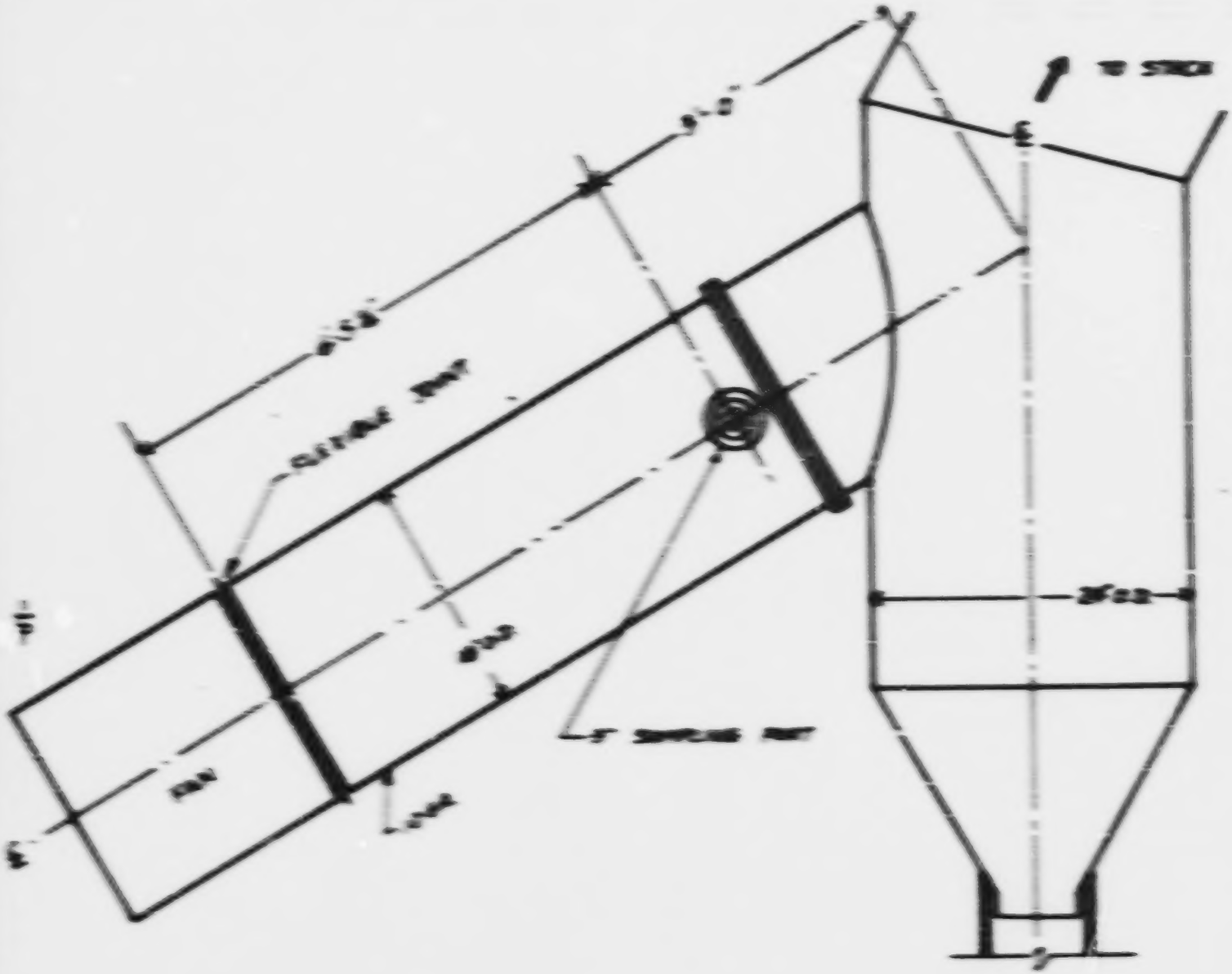


TABLE 1

[redacted]

Location	Hole #	Physical Position*
High Temp. Middle Plot	1	24"
	2	20"
	3	16"
	4	12"
	5	08"
	6	04"
	7	00"
	8	04"
	9	08"
	10	12"
Bleed	1	17"
	2	13"
	3	10"
	4	8"
	5	5"
	6	1"

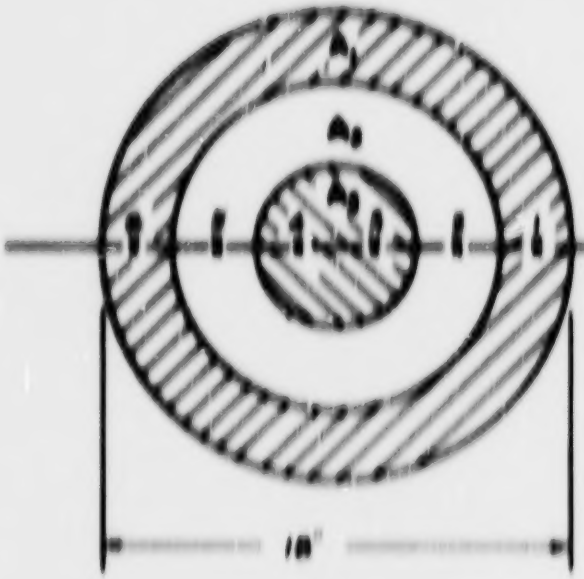
*See Figures 3 and 4 plus body of discussion for detailed explanation of hole location. Measured from nearest inside wall of test trailer section.

TABLE 4

GAS FLOW RATE* FOR PITOT TESTS
Palo Alto Municipal Incinerator

Test No.	Q ft³/min	T °F	Nominal Head in. H_2O	Q ft/min
1	143	300	-.15	74.7
5	139.0	300	-.20	79.1
7	143.9	300	-.20	80.9
9	154.9	300	-.20	87.7
11	153.6	300	-.20	86.4
14	90.4	300	-.04	51.2
16	84.5	300	-.07	47.8
19	119.7	300	-.13	67.7
21	111.3	300	-.13	62.9

*Method of Averaging Velocities



Traverse Idiom

Area A_1 = 128 in², $\bar{v}$ = $\frac{v_1 + v_2}{2}$

Area A_2 = 452 in², $\bar{v}_2$ = $\frac{v_2 + v_3}{2}$

Area A_3 = 4π in², $\bar{v}_3$ = $\frac{v_3 + v_4}{2}$

$$Q(ft^3/min) = \frac{\pi}{144}\left|128\bar{v}_1 + 452\bar{v}_2 + 4\bar{v}_3\right| \text{ where } v \text{ in ft/min}$$

The situation within the afterburner branch is much more complex than that in the exhaust stack. The results of a typical set of velocity pressure/temperature scan across the three accessible ports are shown in Table 5. Since, from Figure 3, it is obvious that considerable turbulence should be found here, it was deemed appropriate to use the vector component character of the double pitot in an attempt to determine the direction of the flow at each traverse sampling point. When one compares the vector sum of the vertical and horizontal components of velocity to that measured at 45° to the vertical, it is clear that the velocity has a component perpendicular to the plane of the pitot assembly - a component which varies with sampling position. The upshot of all this is that the flow rate cannot be simply related to the velocity measurement and so must be obtained indirectly as indicated below. The data presented in Table 5 also show that any attempt to obtain isokinetic sampling is nothing more than a pious hope to convention.

If it is assumed that there are no leaks within the system (afterburner-precooler-scrubber-exhaust) then the mass (dry) of gas passing through the afterburner branch per unit time must equal that passing through the exhaust stack. Then it is possible to compute the flow through the afterburner branch. It is also necessary to include the contribution due to the mass of evaporated water from the drying sludge since some of this water is condensed within the impinger train and thus contributes to the sample.

3.5 Sampling Data - Gas Streams

Summaries of gas sampling data taken during the background and actual sampling runs for both stack and afterburner branch are shown in Table 6 for the afterburner and Table 7 for the stack. The corresponding gas flow rates within the sampled areas are summarized in Table 8.

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[redacted]		[redacted]	[redacted]
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3.0 ANALYTICAL METHODS AND RESULTS

3.1 Analysis

Analysis of all samples for residual PCB was accomplished by the EPA standard method as detailed in Appendix I which consists of extraction with a suitable solvent, column chromatographic cleanup, solvent evaporation, and gas chromatographic analysis. In order to assure uniform precision ^{13}C was frequently used as an internal tracer.

3.2 Analytical Results

The analytical results show that the level of PCB (including all components) is below the level of detection for the ash, scrubber solution and included particulate matter. Only in the air streams was there a significant level of PCB as displayed in Table 9. To repeat, samples 4 and 5 represent background rates at the high temperature breach and the stack, respectively. Samples 6a, 9, 8 and 10 are successive samples taken from the high temperature breach during PCB feed at a rate of 22.5 gm/hr. Similarly, samples 7, 9 and 11 were taken at the stack.

Since no detectable PCB's were found in either the scrubber or the ash, only the exhaust stream need be considered when computing the effectiveness of the multiple hearth furnace in combusting PCB's. The results of the present measurements are indicated in Table 10 where destruction exceeding 99.9 per cent was found in each of the successive tests. Clearly these results are fully in keeping with the earlier results on the combustion of PCB in a similar furnace.

4.0 DISCUSSION

Several interesting features of this experiment can be seen by examination of the data in Table 9. If we compare column 3 with column 4, it is clear that there is little tendency for the PCB's to be carried off with the particulate matter. In fact, during the PCB feed experiments 6a through 11 the PCB concentrations within the particulate matter in either sampling region is, if anything, lower than that seen in the background experiments.

TABLE 9

[illegible]

Test No.	Gas Sample Vol. (scf) (m³)	PCB Collected Impinger (ug)	Filter (ug)	Total PCB (ug)	PCB Concentration (ug/m³)
4	0.175	8.6	7.4	16	91
5	0.747	184.6	4.3	188	252
6A	0.165	422.0	14.1	436	2642
7	0.870	80.1	9.4	90	104
8	0.141	158.6	13.8	172	2601
9	0.946	130.0	0.13	139	147
10	0.139	160.1	0.26	161	1158
11	0.838	43.2	0.11	43	51.7

[illegible]

Test No.	PCB Concentration (ng/m³)	Flue Gas Flow (m³/min)	Exhaust Rate (g/hr)	Feed Rate (g/hr)	Destruction Efficiency (%)*
7	101	3.90	1.224	22.5	94.6
9	147	3.54	1.873	22.5	91.7
11	91.7	3.54	0.663	22.5	97.1

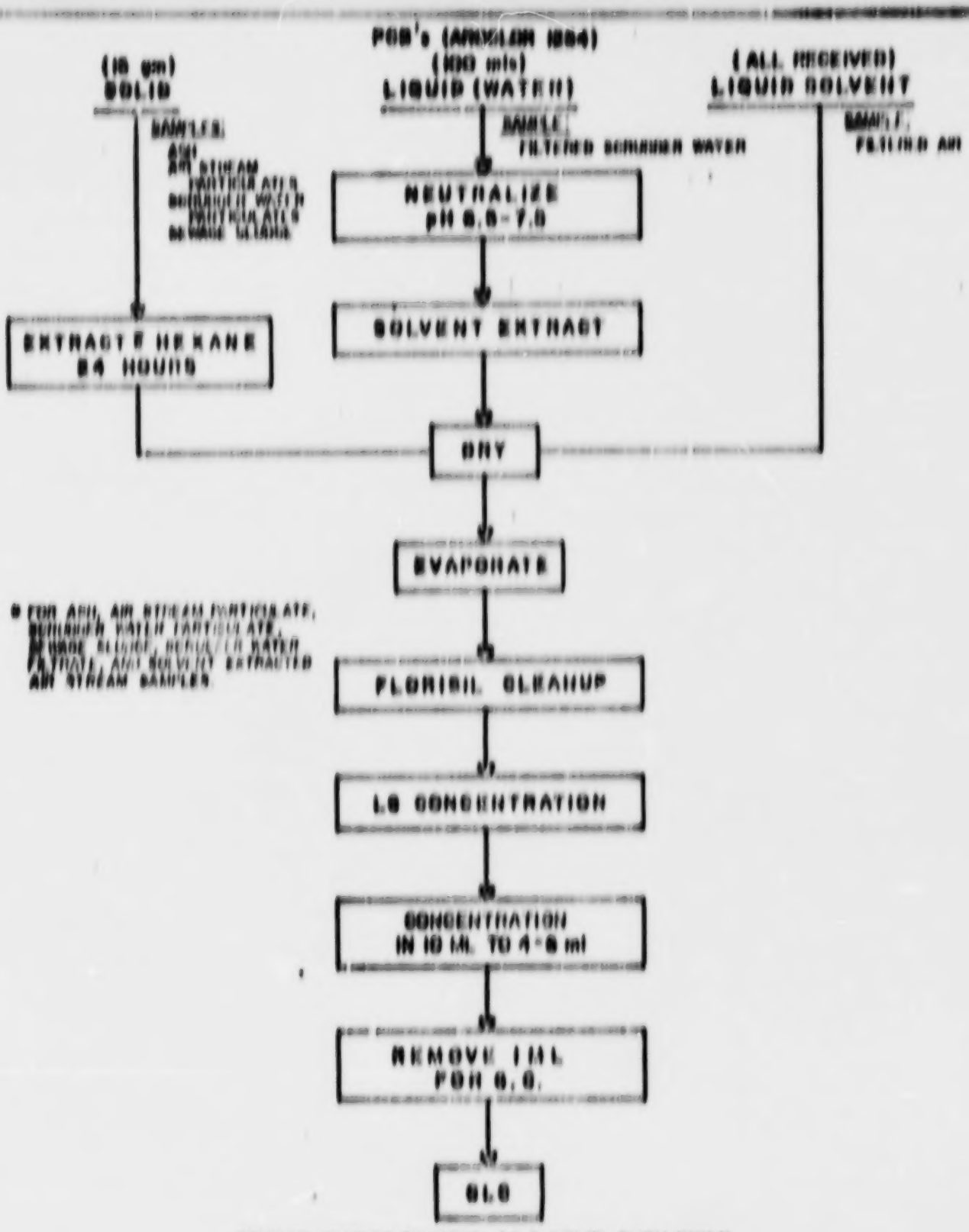
*Since there is no other exhaust stream which carries PCB's, only the stack exhaust must be considered in computing the per cent destruction.

If one compares the total PCB concentration, as shown in column 6, Table 9, it is clear that a great deal of PCB is distilled directly from the drying sludge into the air stream emerging from the afterburner. The fact that the levels within the stack are significantly lower than those within the afterburner breech, coupled with the considerably higher flow rates in the latter region, suggest some mechanism whereby the residual PCB is destroyed while traversing the high temperature regions of the furnace prior to the scrubbing of the hot gases within the scrubber. Apparently, at the temperatures involved (600°-700°C) PCB's are unstable enough to rapidly decay, especially in the presence of high temperature water vapor. From this observation it would appear that in order to further reduce the levels of PCB's within the exhaust stream it would only be necessary to increase the maximum time to this range of temperatures - perhaps simply by increasing the path from the afterburner breech to the scrubber input. Certainly, there appears to be no reason to require significantly higher afterburner temperatures than those present in this experiment. It is also apparent that the scrubber has no effect on the gaseous PCB; considerable improvement could be obtained if a more efficient scrubber system were utilized.

[redacted]

[redacted]

Since a critical part of this experimentation is the reliability with which the analyses have been accomplished, it is most appropriate to include in detail, the analytical method used and to display specific chromatograms representing results obtained in the course of these analyses. It is appropriate to compare the chromatograms which follow with that of the test material shown in Figure 2 of the body of this report. The appendix includes an analytical flow scheme shown as Figure A-1, NIOSH methods for PCB's and selected GC spectra for typical samples.



SEWAGE SLUDGE TREATED AS A SOLID, OVEN DRIED

FIGURE B-1
ANALYTICAL FLOW SCHEME
FOR PCB's (AROCLOR 1254) AND ITS DEGRADATION PRODUCTS*

6. METHOD FOR POLYCHLORINATED BIPHENYLS (PCB'S) IN INDUSTRIAL EFFLUENTS

1. Scope and Application

1.1 This method covers the determination of certain polychlorinated biphenyl (PCB) mixtures including: Aroclors 1221, 1232, 1242, 1248, 1254, 1260 and 1016.

1.2 The method is an extension of the method for organochlorine pesticides in industrial effluents (1). It is designed so that determination of both the PCB's and the organochlorine pesticides may be made on the same sample.

1.3 The limit of detection is approximately 1 µg/l for each Aroclor mixture.

2. Summary

2.1 The PCB's and the organochlorine pesticides are co-extracted by liquid-liquid extraction and, insofar as possible, the two classes of compounds separated from one another prior to gas chromatographic determination. A combination of the standard Florisil column cleanup procedure and a silica gel microcolumn separation procedure (2)(3) are employed. Identification is made from gas chromatographic patterns obtained through the use of two or more unlike columns. Detection and measurement is accomplished using an electron capture, microcoulometric, or electrolytic conductivity detector. Techniques for confirming qualitative identification are suggested.

3. Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Refer

to (4), Part I, Sections 1.4 and 1.5.

3.2 The interferences in industrial effluents are high and varied and pose great difficulty in obtaining accurate and precise measurement of PCB's and organochlorine pesticides. Separation and cleanup procedures are generally required to eliminate these interferences; however, such techniques may result in the loss of certain organochlorine compounds. For this reason great care should be exercised in the selection and use of methods for eliminating or minimizing interferences. It is not possible to describe procedures for overcoming all of the interferences that may be encountered in industrial wastes.

3.3 Phthalate esters, certain organophosphorus pesticides, and elemental sulfur will interfere when using electron capture for detection. These materials do not interfere when the microcoulometric or electrolytic conductivity detectors are used in the halogen mode.

3.4 Organochlorine pesticides and other halogenated compounds constitute interferences in the determination of PCB's. Most of these are separated by the method described below. However, certain compounds, if present in the sample, will occur with the PCB's. Included are: Sulfur, Heptachlor, Aldrin, DDE, technical chlordane, mirex, and to some extent o,p'-DDT and p,p'-DDT.

4. Apparatus and Materials

4.1 Gas Chromatograph - Equipped with glass lined injection port.

4.2 Detector Options:

4.2.1 Electron Capture - Radioactive (tritium or nickel-63)

4.2.2 Microcoulometric Titration

4.2.3 Electrolytic Conductivity

4.3 Recorder - Potentiometric strip chart (10 in.) compatible with detector system.

4.4 Gas Chromatographic Column Materials:

4.4.1 Tubing - Pyrex (180 cm long X 4 mm ID)

4.4.2 Glass Wool - Silanized

4.4.3 Solid Support - Gas-Chrom Q (100-120 mesh)

4.4.4 Liquid Phases - Expressed as weight percent coated on solid support:

4.4.4.1 SE-30 or OV-1, 5%

4.4.4.2 OV-17, 1.5% + QF-1, 1.95%

4.5 Kuderna Danish (K-D) Glassware (Kontes)

4.5.1 Snyder Columns - three ball (macro)

4.5.2 Evaporate Flasks - 500 ml

4.5.3 Receiver Ampuls - 10 ml, graduated

4.5.4 Ampul stoppers

4.6 Chromatographic Column - Chromaflex (400 mm long X 19 mm ID) with coarse fritted plate on bottom and Teflon stopcock; 250 ml reservoir bulb at top of column with flared out funnel shape at top of bulb - a special order (Kontes K-420540-0011).

4.7 Chromatographic Column - Pyrex (approximately 400 mm long X 20 mm ID) with a coarse fritted plate on bottom.

4.8 Micro Column Pyrex - constructed according to Figure 1.

4.9 Capillary pipets disposable (5-3/4 in.) with rubber bulb. (Scientific Products P5205-1).

4.10 Low pressure regulator - 0 to 5 PSIG - with low-flow needle valve (See Figure 1, Matheson Model 70).

4.11 Beaker - 100 ml

4.12 Micro syringes - 10, 25, 50 and 100 µl.

4.13 Separatory Funnels - 125 ml, 1000 ml, and 2000 ml with Teflon stopcocks.

4.14 Graduated Cylinders - 100 ml, 250 ml.

4.15 Blender - High speed, glass or stainless cup.

4.16 Florisil - PR Grade (60-100 mesh); purchase activated at 1250 F and store in the dark in glass containers with glass stopper or foil-lined screw caps. Before use, activate each batch overnight at 130 in foil-covered glass container. Determine lauric acid value (See Appendix I).

4.17 Silica gel - Davison code 950-08-08-236 (60/80 mesh).

4.18 Glass Wool - Hexane extracted.

4.19 Centrifuge Tubes - Pyrex calibrated (15 ml).

5. Reagents, Solvents and Standards

5.1 Ferrous Sulfate - (ACS) 30% solution in distilled water.

5.2 Potassium Iodide - (ACS) 10% solution in distilled water.

5.3 Sodium Chloride - (ACS) Saturated solution (pre-rinse NaCl with hexane) in distilled water.

5.4 Sodium Hydroxide - (ACS) 10 N in distilled water.

5.5 Sodium Sulfate - (ACS) Granular, anhydrous, conditioned for 4 hours @ 400 C.

5.6 Sulfuric Acid - (ACS) Mix equal volumes of conc. H_2SO_4 with distilled water.

5.7 Diethyl Ether - Nanograde, redistilled in glass, if necessary.

5.7.1 Must contain 2% alcohol and be free of peroxides by following test: to 10 ml of ether in glass-stoppered cylinder previously rinsed with ether, add one ml of freshly prepared 10% KI solution. Shake and let stand one minute. No yellow color should be observed in either layer.

5.7.2 Decompose other peroxides by adding 40 g of 30% ferrous sulfate solution to each liter of solvent. CAUTION: Reaction may be vigorous if the solvent contains a high concentration of peroxides.

5.7.3 Distill deperoxidized ether in glass and add 2% ethanol.

5.8 n-Hexane - Pesticide quality (MAY USED MIXED HEXANES).

5.9 Acetonitrile, Hexane, Methanol, Methylene chloride, Petroleum Ether (Boiling range 30-60 C) - pesticide quality, redistill in glass if necessary.

5.10 Standards - Aroclors 1221, 1232, 1242, 1248, 1254, 1260, and 1016.

5.11 Anti-static Solution - STATHOL, Daystrom, Inc., Weston Instrument Division, Newark, N.J. 05212.

6. Calibration

6.1 Gas chromatographic operating conditions are considered acceptable when the response to dieldrin is at least 50% of full scale when 0.06 ng is injected for electron capture detection and 100 ng is injected for microcoulometric or electrolytic conductivity detection. For all quantitative measurements, the detector must be operated within its linear response range and the detector noise level should be less than 5% of full scale.

6.2 Standards are injected frequently as a check on the stability of operating conditions, detector and column. Example chromatograms are shown in Figures 5 through 8 and provide reference operating conditions.

7. Quality Control

7.1 Duplicate and spiked sample analyses are recommended as a quality control check. When the routine occurrence of a pollution parameter is observed, quality control charts are also recommended (5).

7.2 Each time a set of samples is extracted, a method blank is determined on a volume of distilled water equal to that used to dilute the sample.

8. Sample Preparation

8.1 Blend the sample if suspended matter is present and adjust pH to near neutral (pH 6.5-7.5) with 50% sulfuric acid or 10 N sodium hydroxide.

8.2 For a sensitivity requirement of 1 µg/l, when using microcoulometric or electrolytic conductivity methods for detection take 100 ml of sample for analysis. If interferences pose no problem, the sensitivity of the electron capture detector should permit as little as 50 ml of sample to be used. Background information on the extent and nature of interferences will assist the analyst in choosing the required sample size and preferred detector.

8.3 Quantitatively transfer the proper aliquot into a two liter separatory funnel and dilute to one liter.

9. Extraction

9.1 Add 60 ml of 15% methylene chloride in hexane (v:v) to the sample in the separatory funnel and shake vigorously for two minutes.

9.2 Allow the mixed solvent to separate from the sample, then draw the water into a one-liter Erlenmeyer flask. Pass the organic layer through a column containing 3-4 inches of anhydrous sodium sulfate, and collect it in a 500 ml K-D flask equipped with a 10 ml ampul. Return the water phase to the separatory funnel. Rinse the Erlenmeyer flask with a second 60 ml volume of solvent; add the solvent to the separatory funnel and complete the extraction procedure a second time. Perform a third extraction in the same manner.

9.3 Concentrate the extract to 5-10 ml in the K-D evaporator on a hot water bath.

9.4 Qualitatively analyze the sample by gas chromatography with an electron capture detector. From the response obtained decide:

a. If there are any organochlorine pesticides present,

b. If there are any PCB's present,

c. If there is a combination of a and b,

d. If elemental sulfur is present,

e. If the response is too complex to determine a, b, or c.

f. If no response, concentrate to 1.0 ml or less, as required, according to EPA Method (4), pg. 28 and repeat the analysis looking for a, b, c, d, and e. Samples containing Aroclors with a low percentage of chlorine, eg. 1221 and 1232, may require this concentration in order to achieve the detection limit of 1 ug/l. Trace quantities of PCB's are often masked by background which usually occur in the samples.

9.5 If condition a exists, quantitatively determine the organochlorine pesticides according to (1).

9.6 If condition b exists, PCB's only are present, no further separation or cleanup is necessary. Quantitatively determine the PCB's according to 11 below.

9.7 If condition c exists, compare peaks obtained from the sample to those of standard Aroclors and make a judgment as to which Aroclors may be present. To separate the PCB's from the organochlorine pesticides, continue as outlined in 10.4.

9.8 If condition d exists separate the sulfur from the sample using the method outlined in (10.3) followed by the method in (10.5).

9.9 If condition e exists then the following micro cleanup and separation procedures (10.2 and 10.3) should be employed and, if necessary, followed by the micro separation procedures (10.4 and 10.5).

10. Cleanup and Separation Procedures

10.1 Interferences in the form of distinct peaks and/or high background in the initial gas chromatographic analysis, as well as, the physical characteristics of the extract (color, cloudiness, viscosity) and background knowledge of the sample will indicate whether cleanup is required. When these interfere with measurement of the pesticides, or affect column life or detector sensitivity, proceed as directed below.

10.2 Acetonitrile Partition - This procedure is used to remove fats and oils from the sample extracts. It should be noted that not all pesticides are quantitatively recovered by this procedure. The analyst must be aware of this and demonstrate the efficiency of the partitioning for the compounds of interest.

10.2.1 Quantitatively transfer the previously concentrated extract to a 125 ml separatory funnel with enough hexane to bring the final volume to 15 ml. Extract the sample four times by shaking vigorously for one minute with 30 ml portions of hexane-saturated acetonitrile.

10.2.2 Combine and transfer the acetonitrile phases to a one-liter separatory funnel and add 650 ml of distilled water and 40 ml of saturated sodium chloride solution. Mix thoroughly for 30-35 seconds. Extract with two 100 ml portions of hexane by vigorously shaking about 15 seconds.

10.2.3 Combine the hexane extracts in a one-liter separatory funnel and wash with two 100 ml portions of distilled water. Discard the water layer and pour the hexane layer through a 3-4 inch anhydrous sodium sulfate column into a 500 ml K-D

flask equipped with a 10 ml ampul. Rinse the separatory funnel and column with three 10 ml portions of hexane.

10.4.4 Concentrate the extracts to 5-10 ml in the K-D evaporator in a hot water bath.

10.4.5 Analyze by gas chromatography unless a need for further cleanup is indicated.

10.5 Florisil Column Adsorption Chromatography

10.5.1 Adjust the sample extract volume to 10 ml.

10.5.2 Place a charge of activated Florisil (weight determined by lauric acid value, see Appendix I) in a chromatographic column. After settling the Florisil by tapping the column, add about one-half inch layer of anhydrous granular sodium sulfate to the top.

10.5.3 Pre-elute the column, after cooling, with 50-60 ml of petroleum ether. Discard the eluate and just prior to exposure of the sulfate layer to air, quantitatively transfer the sample extract into the column by decantation and subsequent petroleum ether washings. Adjust the elution rate to about 5 ml per minute and, separately, collect up to three eluates in 500 ml K-D flasks equipped with 10 ml ampuls. (See Eluate Composition below). Perform the first elution with 200 ml of 6% ethyl ether in petroleum ether, and the second elution with 200 ml of 15% ethyl ether in petroleum ether. Perform the third elution with 200 ml of 50% ethyl ether : petroleum ether and the fourth elution with 200 ml of 100% ethyl ether.

Eluate Composition - By using an equivalent quantity of any batch of Florisil as determined by its lauric acid value, the pesticides will be separated into the eluates indicated below:

6% Eluate

Aldrin	DDT	Pentachloro-nitrobenzene
BHC	Heptachlor	
Chlordane	Heptachlor epoxide	Nitrofene
DDD	Lindane	Toxaphene
DDE	Methoxychlor	Trifluralin
	Mirex	PCB's

15% Eluate

Endosulfan I
Endrin
Dieldrin
Dichloran
Phthalate esters

50% Eluate

Endosulfan II
Captan

Certain thiophosphate pesticides will occur in each of the above fractions as well as the 100% fraction. For additional information regarding eluate composition, refer to the FDA Pesticide Analytical Manual (5).

10.3.4 Concentrate the eluates to 5-10 ml in the K-D evaporator in a hot water bath.

10.3.5 Analyze by gas chromatography.

10.4 Silica Gel Micro-Column Separation Procedure (7)

10.4.1 Activation for Silica Gel

10.4.1.1 Place about 20 gm of silica gel in a 100 ml beaker. Activate at 150 C for approximately 16 hours. Transfer the silica gel to a 100 ml glass stoppered bottle. When cool, cover with about 35 ml of 0.50% diethyl ether in benzene (volume:volume). Keep bottle well sealed. If silica gel collects on the ground glass surfaces, wash off with the above solvent

before resealing. Always maintain an excess of the mixed solvent in bottle (approximately 1/2 in. above silica gel). Silica gel can be effectively stored in this manner for several days.

10.4.2 Preparation of the Chromatographic Column

10.4.2.1 Pack the lower 2 mm ID section of the microcolumn with glass wool. Permanently mark the column 100 mm above the glass wool. Using a clean rubber bulb from a disposable pipet seal the lower end of the microcolumn. Fill the microcolumn with 0.50% ether in benzene (v:v) to the bottom of the 10/50 joint (Figure 1). Using a disposable capillary pipet, transfer several aliquots of the silica gel slurry into the microcolumn. After approximately 1 cm of silica gel collects in the bottom of the microcolumn, remove the rubber bulb seal, tap the column to insure that the silica gel settles uniformly. Carefully pack column until the silica gel reaches the 100 ± 2 mm mark. Be sure that there are no air bubbles in the column. Add about 10 mm of sodium sulfate to the top of the silica gel. Under low humidity conditions, the silica gel may coat the sides of the column and not settle properly. This can be minimized by wiping the outside of the column with an anti-static solution.

10.4.8.8 Deactivation of the Silica Gel

a. Fill the microcolumn to the base of the 10/30 joint with the 0.50% ether-benzene mixture, assemble reservoir (using spring clamps) and fill with approximately 15 ml of the 0.50% ether-benzene mixture. Attach the air pressure device (using spring clamps) and adjust the elution rate to approximately 1 ml/min with the air pressure control. Release the air pressure and detach reservoir just as the last of the solvent enters the sodium sulfate. Fill the column with n-hexane (not mixed hexanes) to the base of the 10/30 fitting. Evaporate all residual benzene from the reservoir, reassemble the reservoir section and fill with 5 ml of n-hexane. Apply air pressure and adjust the flow to 1 ml/min. (The n-hexane flows slightly faster than the benzene). Release the air pressure and remove the reservoir just as the n-hexane clears the sodium sulfate. The column is now ready for use.

b. Pipet a 1.0 ml aliquot of the concentrated sample extract (previously reduced to a total volume of 2.0 ml) on to the column.

As the last of the sample passes into the sodium sulfate layer, rinse down the internal wall of the column twice with 0.25 ml of n-hexane. Then assemble the upper section of the column. As the last of the n-hexane rinse reaches the surface of the sodium sulfate, add enough n-hexane (volume predetermined, see 10.4.3 below) to just elute all of the PCB's present in the sample. Apply air pressure and adjust until the flow is 1 ml/min. Collect the desired volume of eluate (predetermined, see 10.4.3 below) in an accurately calibrated ampul. As the last of the n-hexane reaches the surface of the sodium sulfate, release the air pressure and change the collection ampul.

e. Fill the column with 0.50% diethyl ether in benzene, again apply air pressure and adjust flow to 1 ml/min. Collect the eluate until all of the organochlorine pesticides of interest have been eluted (volume predetermined, see 10.4.3 below).

d. Analyze the eluates by gas chromatography.

10.4.3 Determination of Elution Volumes

10.4.3.1 The elution volumes for the PCB's and the pesticides depend upon a number of factors which

are difficult to control. These include variation in:

a. Mesh size of the silica gel
b. Adsorption properties of the silica gel
c. Polar contaminants present in the eluting solvent
d. Polar materials present in the sample and sample solvent
e. The dimensions of the microcolumns

Therefore, the optimum elution volume must be experimentally determined each time a factor is changed. To determine the elution volumes, add standard mixtures of Aroclors and pesticides to the column and serially collect 1 ml elution volumes. Analyse the individual eluates by gas chromatography and determine the cut-off volume for n-hexane and for ether-benzene. Figure 3 shows the retention order of the various PCB components and of the pesticides. Using this information, prepare the mixtures required for calibration of the microcolumn.

10.4.3.2 In determining the volume of hexane required to elute the PCB's the sample volume (1 ml) and the volume of n-hexane used to rinse the column wall must be considered. Thus, if it is determined that a 10.0 ml elution volume is required to elute the PCB's, the volume of hexane to be added

in addition to the sample volume but including the rinse volume should be 0.5 ml.

10.4.3.3 Figure 9 shows that as the average chlorine content of a PCB mixture decreases the solvent volume for complete elution increases. Qualitative determination (9.4) indicates which Aroclors are present and provides the basis for selection of the ideal elution volume. This helps to minimize the quantity of organochlorine pesticides which will elute along with the low percent chlorine PCB's and insures the most efficient separations possible for accurate analysis.

10.4.3.4 For critical analysis where the PCB's and pesticides are not separated completely, the column should be accurately calibrated according to (10.4.3.1) to determine the percent of material of interest that elutes in each fraction. Then flush the column with an additional 15 ml of 0.50% ether in benzene followed by 5 ml of n-hexane and use this reconditioned column for the sample separation. Using this technique one can accurately predict the amount (%) of materials in each micro column fraction.

10.5 Micro Column Separation of Sulfur, PCB's, and Pesticides

10.5.1 See procedure for preparation and packing micro columns in PCB analysis section (10.4.1 and 10.4.2).

10.5.2 Microcolumn Calibration

10.5.2.1 Calibrate the microcolumn for sulfur and PCB separation by collecting 1.0 ml fractions and analysing them by gas chromatography to determine the following:

1) The fraction with the first eluting PCB's (those present in 1260),
2) The fraction with the last eluting PCB's (those present in 1221),
3) The elution volume for sulfur,
4) The elution volume for the pesticides of interest in the 0.50% ether-benzene fraction.

From these data determine the following:

1) The eluting volume containing only sulfur (Fraction I),
2) The eluting volume containing the last of the sulfur and the early eluting PCB's (Fraction II),
3) The eluting volume containing the remaining PCB's (Fraction III),
4) The ether-benzene eluting volume containing the pesticides of interest (Fraction IV).

10.5.3 Separation Procedure

10.5.3.1 Carefully concentrate the 6% eluate from the florisil column to 2.0 ml in the graduated ampul on a warm water bath.

10.5.3.2 Place 1.0 ml (50%) of the concentrate into the microcolumn with a 1 ml pipet. Be careful

not to get any sulfur crystals into the pipet.

10.5.3.3 Collect Fractions I and II in calibrated centrifuge tubes.

Collect Fractions III and IV in calibrated ground glass stoppered ampules.

10.5.3.4 Sulfur Removal (8) - Add 1 to 2 drops of mercury to Fraction II stopper and place on a wrist-action shaker. A black precipitate indicates the presence of sulfur. After approximately 20 minutes the mercury may become entirely reacted or deactivated by the precipitate. The sample should be quantitatively transferred to a clean centrifuge tube and additional mercury added. When crystals are present in the sample, three treatments may be necessary to remove all the sulfur. After all the sulfur has been removed from fraction II (check using gas chromatography) combine Fractions II and III. Adjust the volume to 10 ml and analyse gas chromatography. Be sure no mercury is transferred to the combined fractions II and III, since it can react with certain pesticides.

By combining fractions II and III, if PCB's are present, it is possible to identify the Aroclor(s) present and a quantitative analysis can be performed accordingly. Fraction I can be discarded since it only contains the bulk of the sulfur. Analyse fractions III and IV for the PCB's and

pesticides. If DDT and its homologs, aldrin, heptachlor, or technical chlordane are present along with the PCB's, an additional micro-column separation can be performed which may help to further separate the PCB's from the pesticides (Sec 10.4).

11. Quantitative Determination

11.1 Measure the volume of n-hexane eluate, containing the PCB's and inject 1 to 5 µl into the gas chromatograph. If necessary, adjust the volume of the eluate to give linear response to the electron capture detector. The microcoulometric or the electrolytic detector may be employed to improve specificity for samples having higher concentrations of PCB's.

11.2 Calculations

11.2.1 When a single Aroclor is present, compare quantitative Aroclor reference standards (e.g., 1242, 1260) to the unknown. Measure and sum the areas of the unknown and the reference Aroclor and calculate the result as follows:

$$\text{Microgram/liter} = \frac{(A)\,(B)\,(V_t)}{(V_i)\,(V_s)} \times (N)$$

$A = \frac{\text{ng of Standard Injected}}{\text{Σ of Standard Peak Areas}} = \frac{ng}{mm^2}$

$B = \Sigma$ of Sample Peak Areas $= (mm^2)$

V_i = Volume of sample injected (µl)

V_t = Volume of Extract (µl) from which sample is injected into gas chromatograph

V_s = Volume of water sample extracted (ml)

N = 2 when micro column used
1 when micro column not used

Peak Area = Peak height (mm) x Peak Width at 1/2 height

11.2.2 For complex situations, use the calibration method described below. Small variations in components between different Aroclor batches make it necessary to obtain samples of several specific Aroclors. These reference Aroclors can be obtained from Dr. Ronald Webb, Southeast Environmental Research Laboratory, EPA, Athens, Georgia 30601. The procedure is as follows:

11.2.2.1 Using the OV-1 column, chromatograph a known quantity of each Aroclor reference standard. Also chromatograph a sample of p,p'-DDE. Suggested concentration of each standard is 0.1 ng/µl for the Aroclors and 0.02 ng/µl for the p,p'-DDE.

11.2.2.2 Determine the relative retention time (RRT) of each PCB peak in the resulting chromatograms using p,p'-DDE as 100. See Figures 3 through 5.

$$RRT = \frac{RT_x}{RT_{DDE}} \times 100$$

RRT = Relative Retention Time

RT = Retention time of peak of interest

RT_{DDE} = Retention time of p,p'-DDE

Retention time is measured as that distance in mm between the first appearance of the solvent peak and the maximum for the compound.

11.2.2.3 To calibrate the instrument for each PCB measure the area of each peak.

Area = Peak height (mm) x Peak width at 1/2 height. Using Tables 1 through 6 obtain the proper mean weight factor, then determine the response factor ng/mm^2.

$$ng/mm^2 = \frac{(ng_1)\ (\text{mean weight percent})}{100\ (\text{Area})}$$

ng_1 = ng of Aroclor Standard injected

Mean weight percent = obtained from tables 1 through 6.

11.9.9.4 Calculate the RRT value and the area for each PCB peak in the sample chromatogram. Compare the sample chromatogram to those obtained for each reference Aroclor standard. If it is apparent that the PCB peaks present are due to only one Aroclor then calculate the concentration of each PCB using the following formula:

ng PCB = ng/mm^2 x Area

Where Area = Area (mm^2) of sample peak

ng/mm^2 = Response factor for that peak measured.

Then add the nanograms of PCB's present in the injection to get the total number of nanograms of PCB's present. Use the following formula to calculate the concentration of PCB's in the sample:

$$\text{Micrograms/liter} = \frac{[ng]\ [V_t]}{[V_s]\ [V_i]} = [N]$$

V_s = volume of water extracted (ml)

V_t = volume of extract (µl)

V_i = volume of sample injected (µl)

ng = sum of all the PCB's in nanograms for that Aroclor identified

N = 2 when microcolumn used

N = 1 when microcolumn not used

The value can then be reported as Micrograms/Liter PCB's reported as the Aroclor. For samples containing more than one Aroclor, use Figure 8 chromatogram divisional flow chart to assign a proper response factor to each peak and also identify the "most likely" Aroclors present. Calculate the ng of each PCB isomer present and sum them according to the divisional flow chart. Using the formula above, calculate the concentration of the various Aroclors present in the sample.

12. Reporting Results

12.1 Report results in micrograms per liter without correction for recovery data. When duplicate and spiked samples are analyzed, all data obtained should be reported.

Table 1

Composition of Aroclor 1221 (8)

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	Number of Chlorines[c]
11	31.6	15.8	1
14	19.3	9.1	1
16	10.1	9.7	2
19	3.8	9.7	2
21	20.0	9.3	2
28	5.4	13.9	2 } 85% 3 } 15%
32	1.4	30.1	2 } 10% 3 } 90%
37 40	1.7	48.8	3 3
Total	93.3		

[a]Retention time relative to p,p'-DDE-13C. Measured from first appearance of solvent. Overlapping peaks that are quantitated as one peak are bracketed.

[b]Standard deviation of seventeen results as a percentage of the mean of the results.

[c]From GC-MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

Table I

Composition of Aroclor 1242 (A)

Area	Mean Weight Percent	Relative Std. Dev.[b]	Number of Chlorines[c]
11	16.8	3.4	1
14	9.0	5.5	1
16	7.1	6.0	2
20	17.0	2.4	2
21			2
28	9.6	3.4	2 40%
			3 60%
32	3.9	4.7	3
37	6.8	2.5	3
40	6.4	2.7	3
47	4.2	4.1	4
54	3.4	3.4	3 33%
			4 67%
58	2.0	3.7	4
70	4.6	3.1	4 90%
			5 10%
78	1.7	7.5	4
Total	94.2		

[a] Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent. Overlapping peaks that are quantitated as one peak are bracketed.

[b] Standard deviation of four results as a mean of the results.

[c] From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

Table 1

Composition of Aroclor 1242 (B)

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	Number of Chlorines[c]
11	1.1	35.7	1
16	2.9	4.2	2
21	11.3	3.0	2
26	11.0	5.6	2 } 25%
			3 } 75%
32	0.1	4.7	3
37	11.5	5.7	3
40	11.1	6.0	3
47	0.8	4.3	4
54	6.8	2.0	3 } 33%
			4 } 67%
58	5.6	3.3	4
70	10.3	2.8	4 } 50%
			5 } 50%
78	3.6	4.2	4
84	2.7	9.7	5
90	1.9	9.4	5
104	2.3	16.4	5
125	1.6	20.4	5 } 85%
			6 } 15%
146	1.0	19.0	5 } 75%
			6 } 25%
Total	98.5		

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.

[b]Standard deviation of six results as a percentage of the mean of the results.

[c]From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

Table 4

Composition of Aroclor 1248 (B)

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	Number of Chlorines[c]
21	1.8	21.9	3
28	5.2	3.3	3
32	3.2	3.8	3
37	0.3	3.6	3
40	0.3	3.0	3 } 85% 4 } 15%
47	15.6	1.1	4
54	9.7	6.0	3 } 10% 4 } 90%
58	9.3	5.8	4
70	10.0	1.4	4 } 80% 5 } 20%
78	6.6	2.7	4
84	4.9	2.6	5
88	3.2	3.2	5
104	3.1	3.6	4 } 10% 5 } 90%
112	1.2	6.6	5
125	2.6	5.9	5 } 90% 6 } 10%
146	1.5	10.0	5 } 85% 6 } 15%
Total	103.1		

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.

[b]Estimated standard deviation of mean weight as a percentage of the mean of the results.

[c]From mass data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

Table 5

Composition of Aroclor 1254 (5)

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	Number of Chlorines
47	0.2	3.7	4
54	2.9	2.6	4
68	1.4	2.0	4
70	13.2	3.7	4] 25%
			5] 75%
84	17.3	1.9	5
88	7.5	6.3	5
101	12.5	3.9	5
126	16.0	2.4	5] 70%
			6] 30%
146	10.4	3.7	5] 70%
			6] 30%
160	1.3	9.4	6
174	8.4	5.5	6
201	1.8	10.6	6
232	1.0	26.1	7
Total	100.0		

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.

[b]Standard deviation of six results as a percentage of the mean of the results.

[c]From GC-MS data. Peaks containing mixtures of isomers are bracketed.

Table 6

Composition of Aroclor 1260 (5)

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	Number of Chlorines[c]
70	0.7	0.3	5
84	4.7	1.6	5
98	9.6	3.5	5 } [d]
104			6 } 60%
			6 } 40%
117	0.3	0.7	6
126	12.1	3.3	6 } 15%
			6 } 85%
146	14.1	3.6	6
160	4.6	2.2	6 } 50%
			7 } 50%
174	12.4	2.7	6
203	6.3	4.0	6 } 10%
			7 } 90%
232			[e]
244	9.8	3.4	6 } 10%
			7 } 90%
280	11.0	2.4	7
338	4.2	5.0	7
372	4.0	8.6	8
440	.6	25.3	8
520	1.5	10.2	8
Total	88.6		

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent. Overlapping peaks that are quantified as one peak are bracketed.

[b]Standard deviation of six results as a mean of the results.

[c]From GC-MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

[d]Composition determined at the center of peak 104.

[e]Composition determined at the center of peak 232.

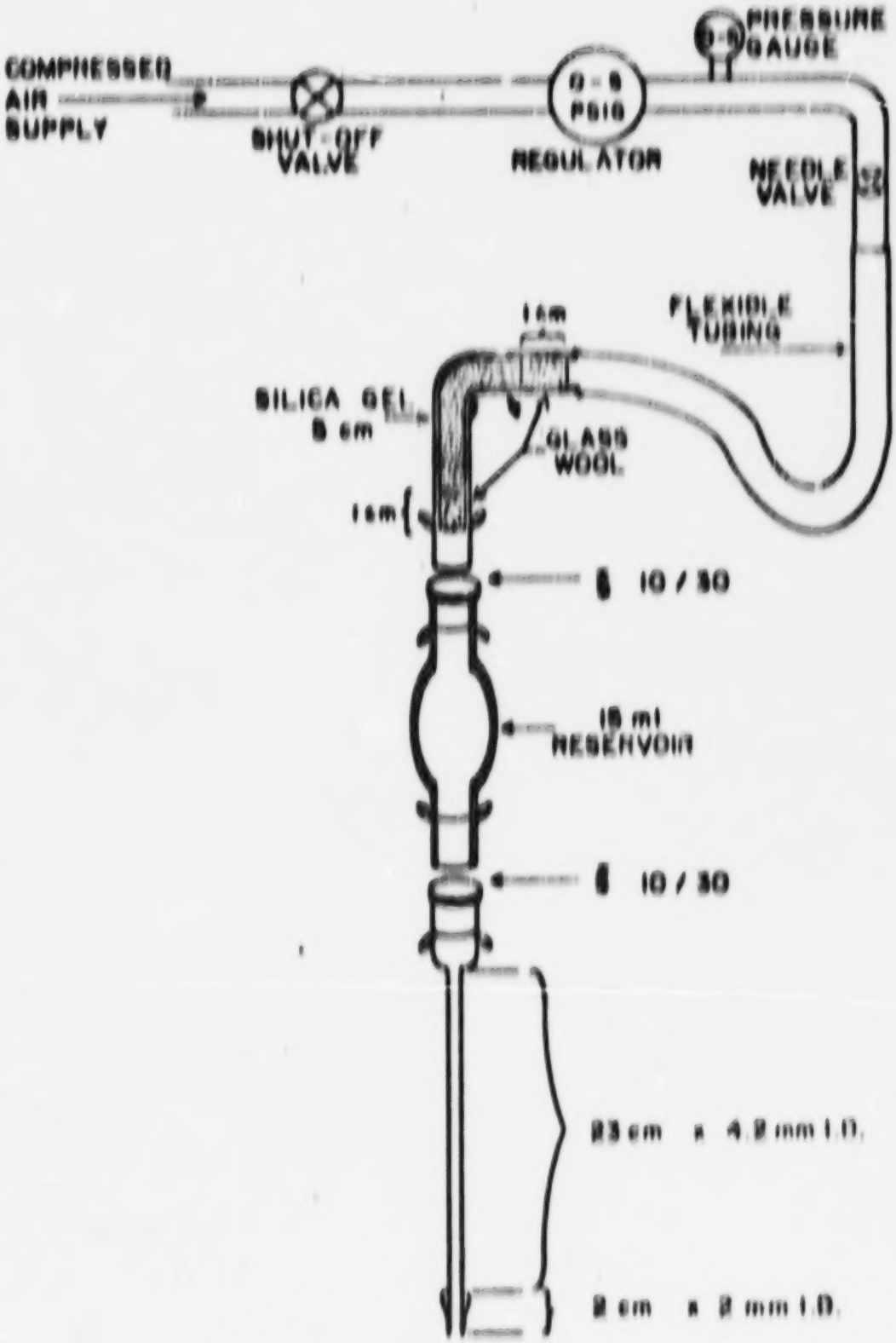


FIGURE 1. MICROCOLUMN SYSTEM

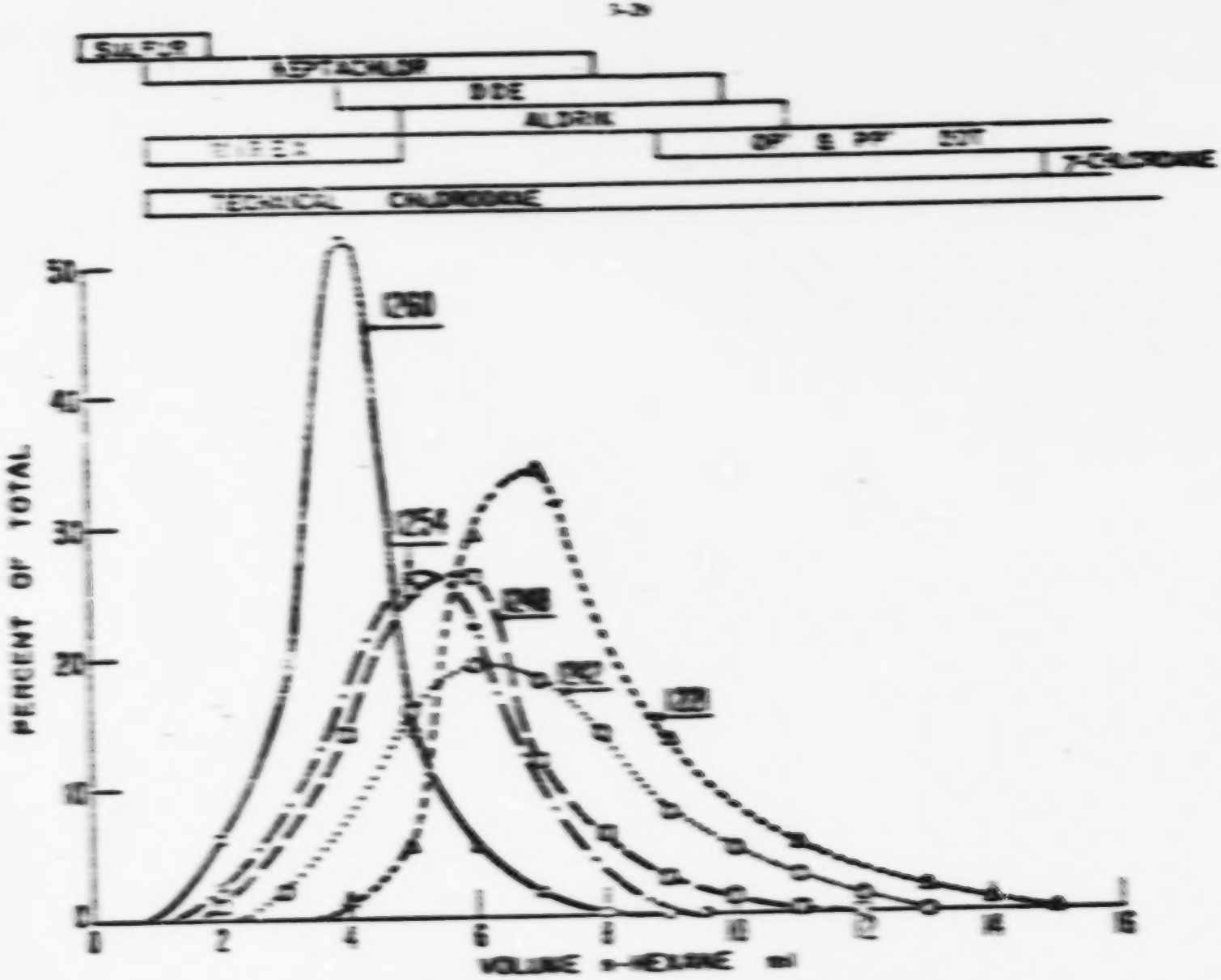


Figure 2. Aroclor Elution Patterns

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APPENDIX I

15. Standardisation of Florisil Column by Weight Adjustment Based on Adsorption of Lauric Acid.

15.1 A rapid method for determining adsorptive capacity of Florisil is based on adsorption of lauric acid from hexane solution (6) (8). An excess of lauric acid is used and amount not adsorbed is measured by alkali titration. Weight of lauric acid adsorbed is used to calculate, by simple proportion, equivalent quantities of Florisil for batches having different adsorptive capacities.

15.2 Apparatus

15.2.1 Buret. -- 25 ml with 1/10 ml graduations.

15.2.2 Erlenmeyer flasks. -- 125 ml narrow mouth and 25 ml, glass stoppered.

15.2.3 Pipet. -- 10 and 20 ml transfer.

15.2.4 Volumetric flasks. -- 500 ml.

15.3 Reagents and Solvents

15.3.1 Alcohol, ethyl. -- USP or absolute, neutralized to phenolphthalein.

15.3.2 Hexane. -- Distilled from all glass apparatus.

15.3.3 Lauric acid. --Purified, CP.

15.3.4 Lauric acid solution. -- Transfer 10.000 g lauric acid to 500 ml volumetric flask, dissolve in hexane, and dilute to 500 ml (1 ml = 20 mg).

15.3.5 Phenolphthalein indicator. -- Dissolve 1 g in alcohol and dilute to 100 ml.

13.3.6 Sodium hydroxide, 1N - Dissolve 20 g NaOH (pellets, reagent grade) in water and dilute to 500 ml (1N). Dilute 25 ml 1N NaOH to 500 ml with water (0.05N). Standardize as follows: Weigh 100-200 mg lauric acid into 125 ml Erlenmeyer flask. Add 50 ml neutralized ethyl alcohol and 3 drops phenolphthalein indicator; titrate to permanent end point. Calculate mg lauric acid/ml 0.05 N NaOH (about 10 mg/ml).

13.4 Procedure

13.4.1 Transfer 2.000 g Florisil to 25 ml glass stoppered Erlenmeyer flasks. Cover loosely with aluminum foil and heat overnight at 130°C. Stopper, cool to room temperature, add 20.0 ml lauric acid solution (400 mg), stopper, and shake occasionally for 15 min. Let adsorbent settle and pipet 10.0 ml of supernatant into 125 ml Erlenmeyer flask. Avoid inclusion of any Florisil.

13.4.2 Add 50 ml neutral alcohol and 3 drops indicator solution; titrate with 0.05N to a permanent end point.

13.5 Calculation of Lauric Acid Value and Adjustment of Column Weight

13.5.1 Calculate amount of lauric acid adsorbed on Florisil as follows:

Lauric Acid value = mg lauric acid/g Florisil = 200 - (ml required for titration X mg lauric acid/ml 0.05N NaOH).

13.5.2 To obtain an equivalent quantity of any batch of Florisil, divide 110 by lauric acid value for that batch and multiply by 20 g. Verify proper elution of pesticides by 13.6.

14.6 Test for Proper Elution Pattern and Recovery of Pesticides: Prepare a test mixture containing aldrin, heptachlor epoxide, p,p'-DDE, dieldrin, Parathion and malathion. Dieldrin and Parathion should elute in the 15% eluate; all but a trace of malathion in the 50% eluate and the others in the 6% eluate.

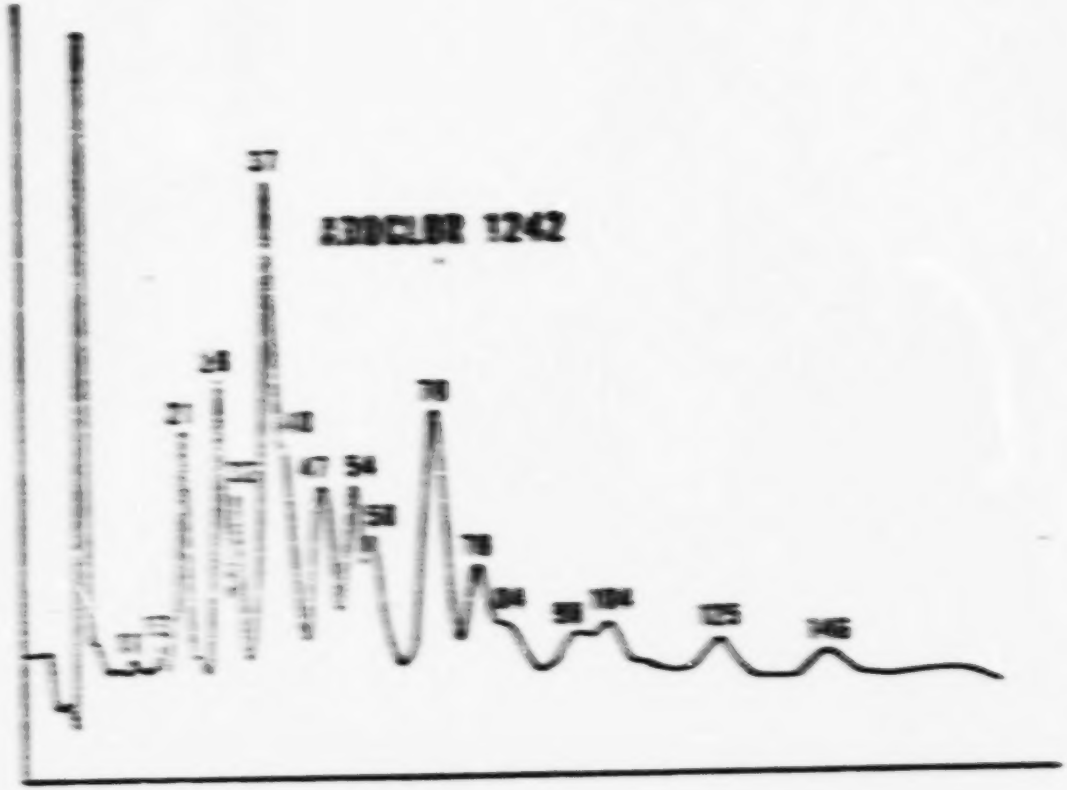

Figure 3. Column: 3% SP-1, Carrier Gas: Nitrogen at 60 ml/min.
Column Temperature: 170 C, Detector: Electron Capture

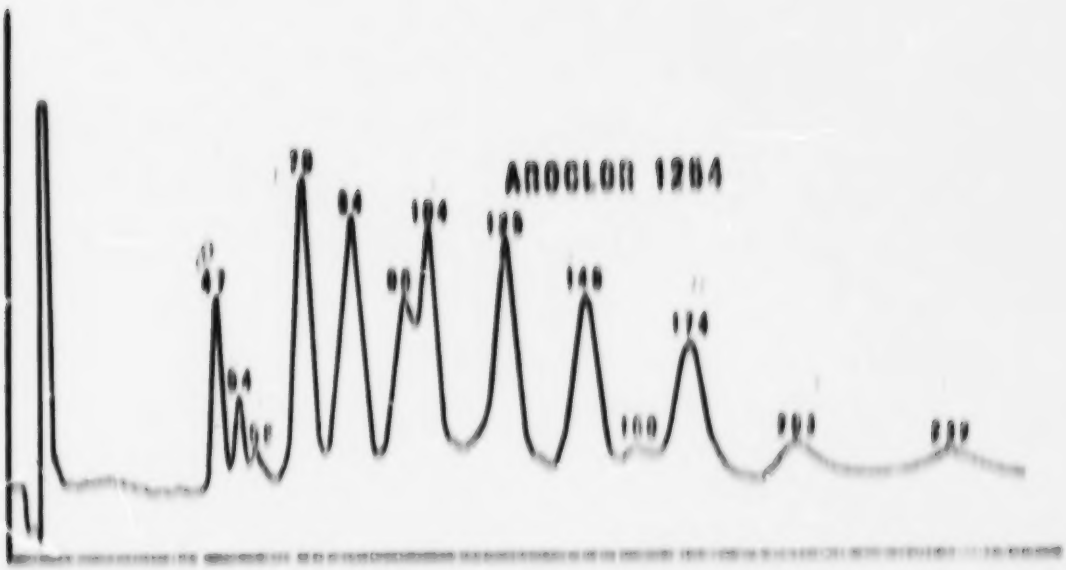

Figure 4. Column: 3% OV-1, Carrier Gas: Nitrogen at 60 ml/min, Column Temperature: 170 C, Detector: Electron Capture.

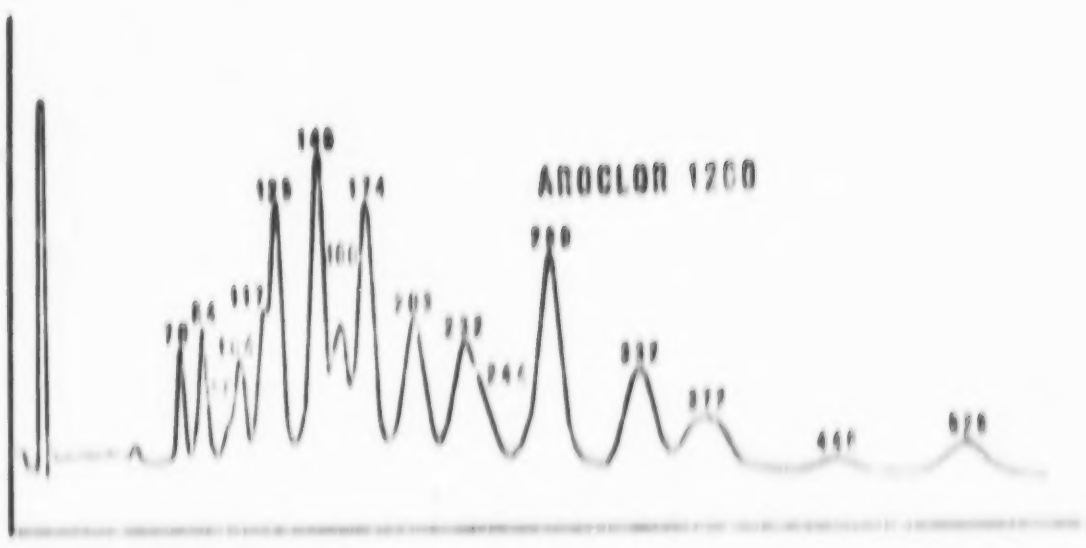

Figure 5. Column: 3% OV-1 Carrier Gas: Nitrogen at 60 ml/min, Column Temperature: 170 C, Detector: Electron Capture

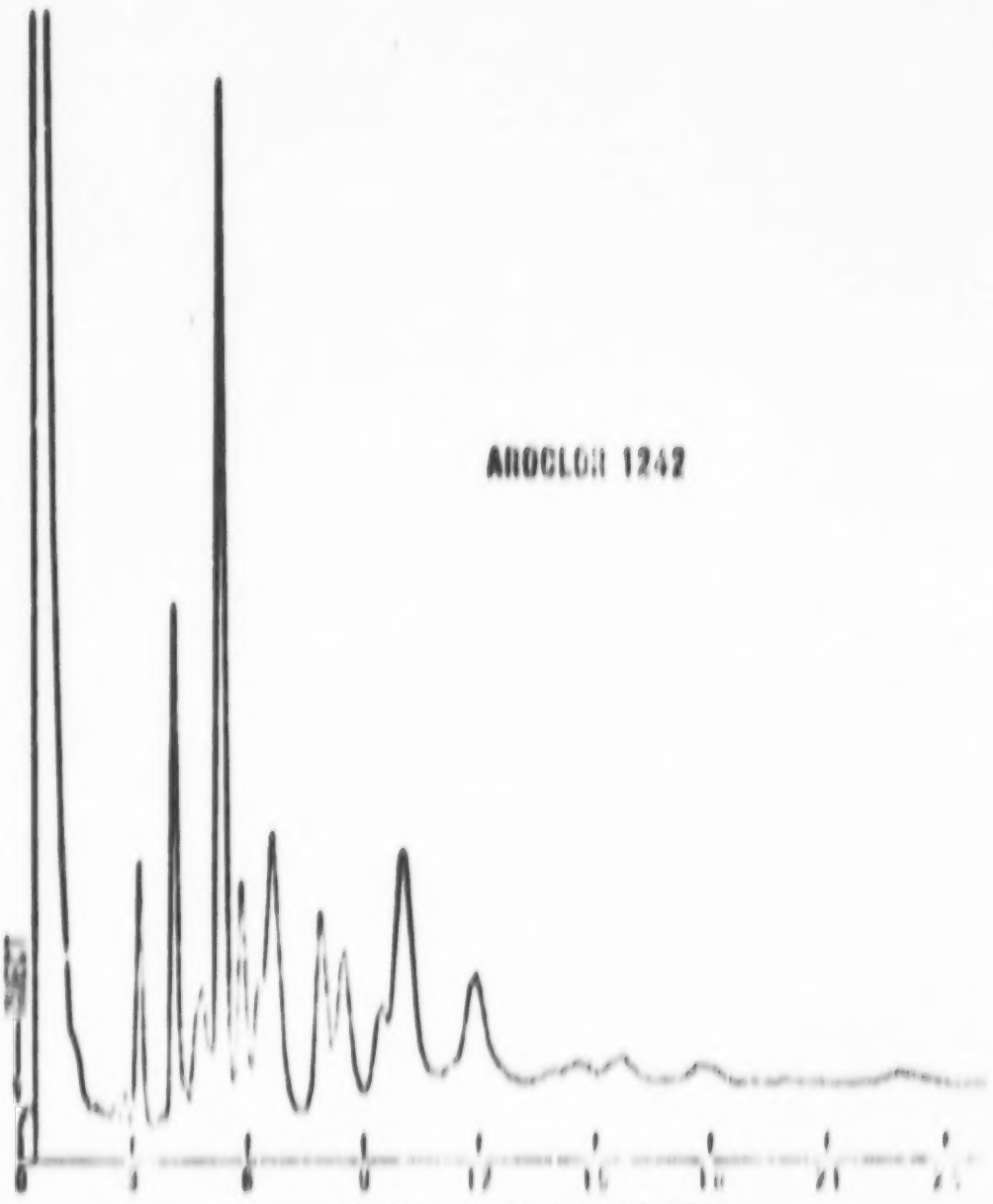

Figure 6. Column: 1.5% OV-17 + 1.95% QF-1, Carrier Gas: Nitrogen at 60 ml/min, Column Temperature: 200 C, Detector: Electron Capture

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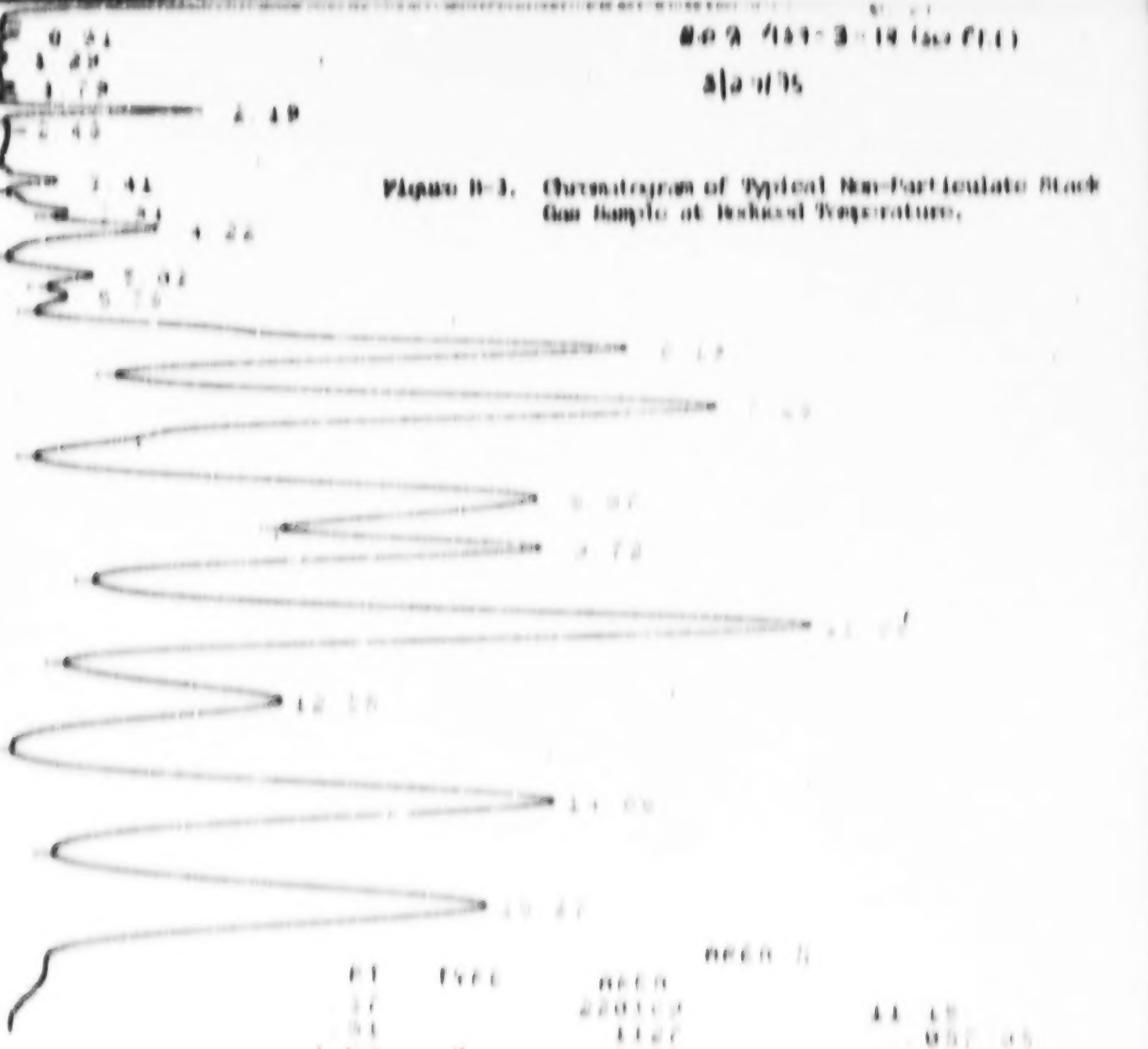
Figure B-2. Chromatogram of Typical High Temperature Heat Gas-Particle Sample.

Figure B-3. Chromatogram of Typical Non-Particulate Stack Gas Sample at Reduced Temperature.



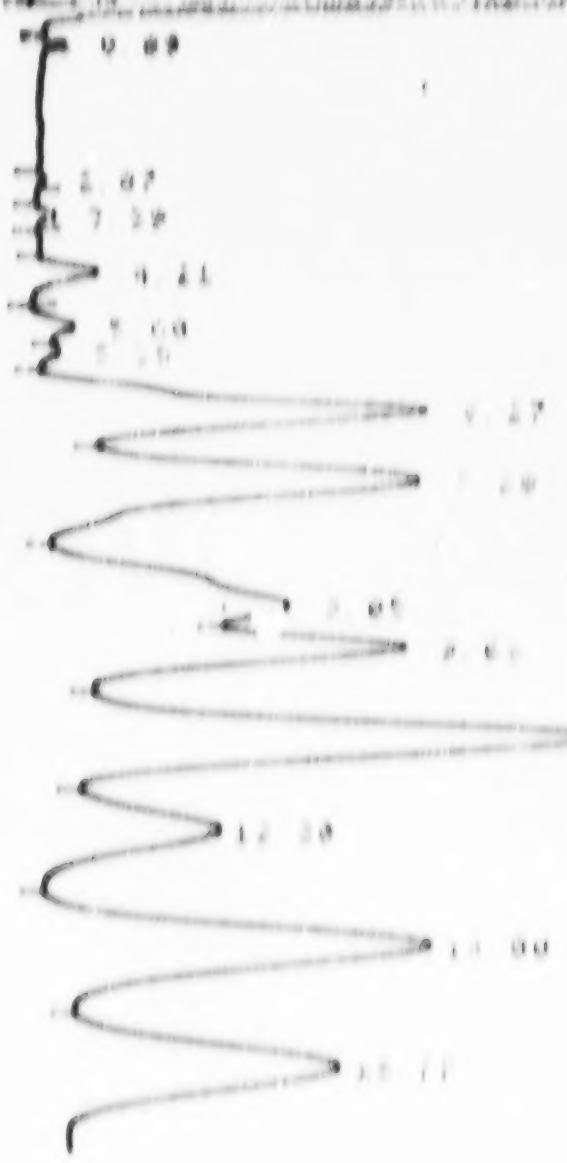


Figure B-4. Chromatogram of Typical High Temperature Test Run Particulate Sample at Reduced Temperature.

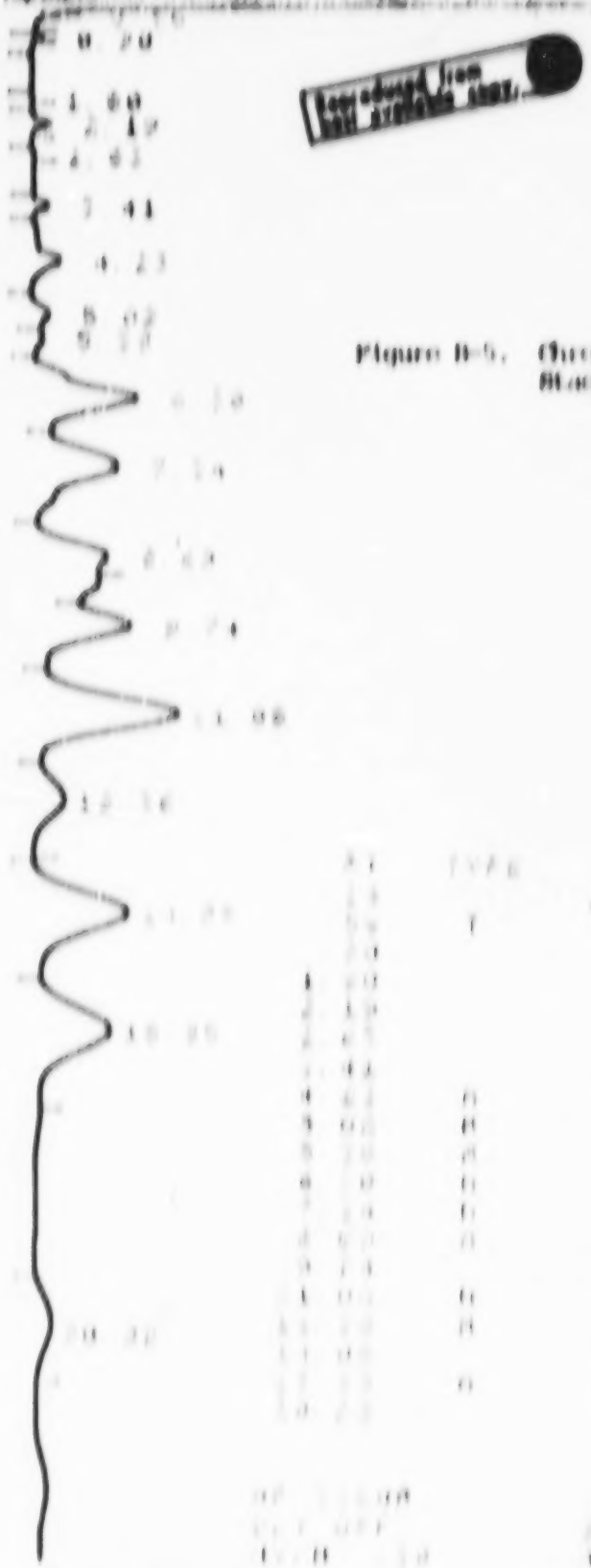


Figure B-5. Chromatogram of Typical Non-Particulate Blank Gas Sample.

Figure B-6. Chromatogram of Particulate Black Gas Sample.

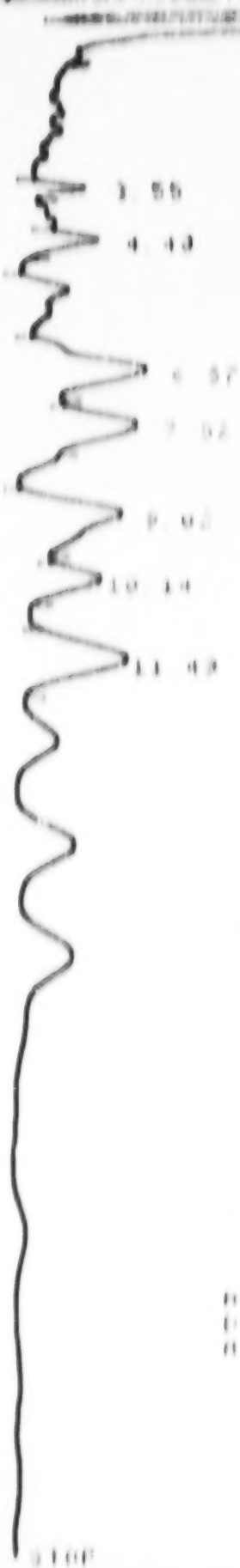


Figure B-7. Chromatogram of High Temperature Test Particulate Sample at Reheat Temperature.

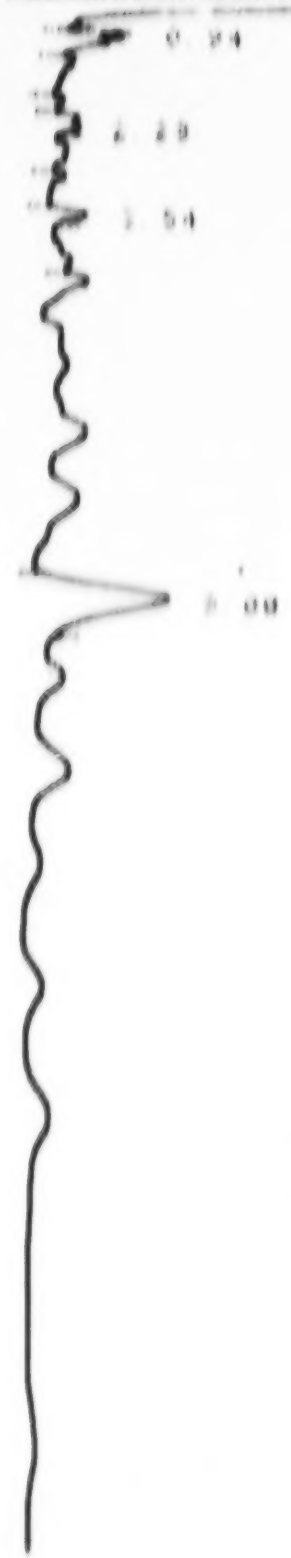


Figure D-8. Chromatogram of High Temperature Port Particulate Sample at Reduced Temperature.

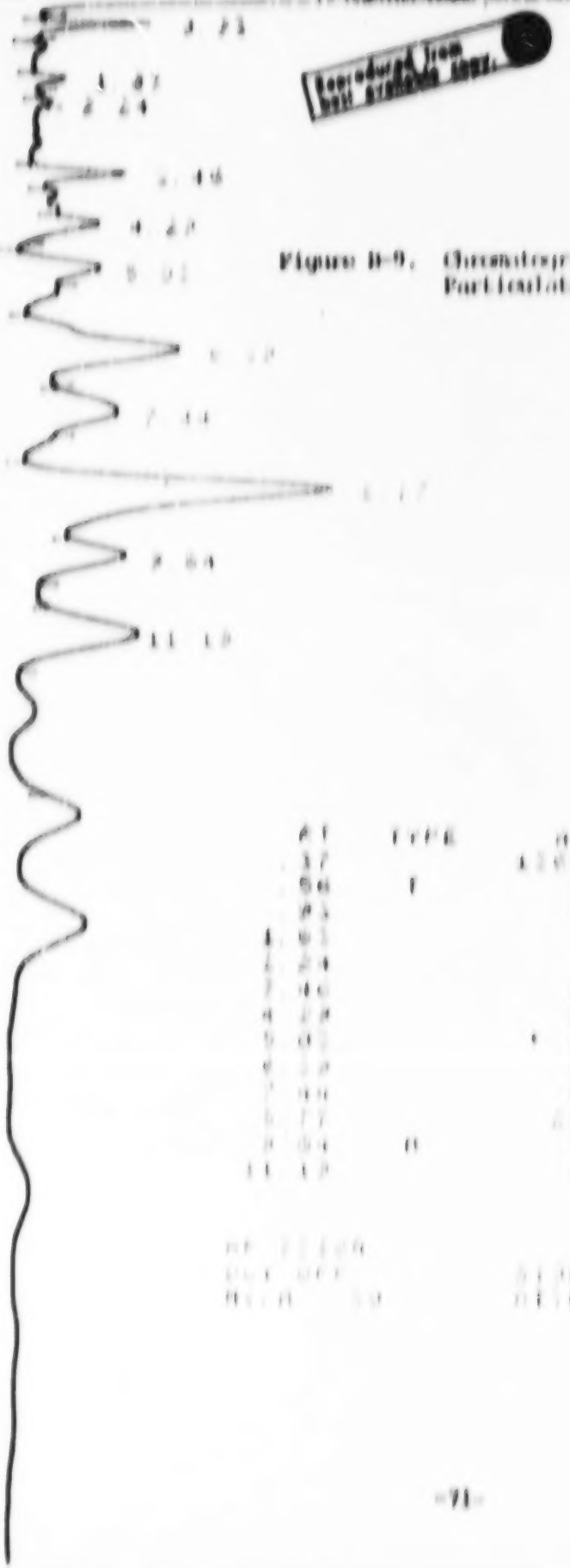


Figure B-9. Chromatogram of Typical High Temperature Particulate Filter.

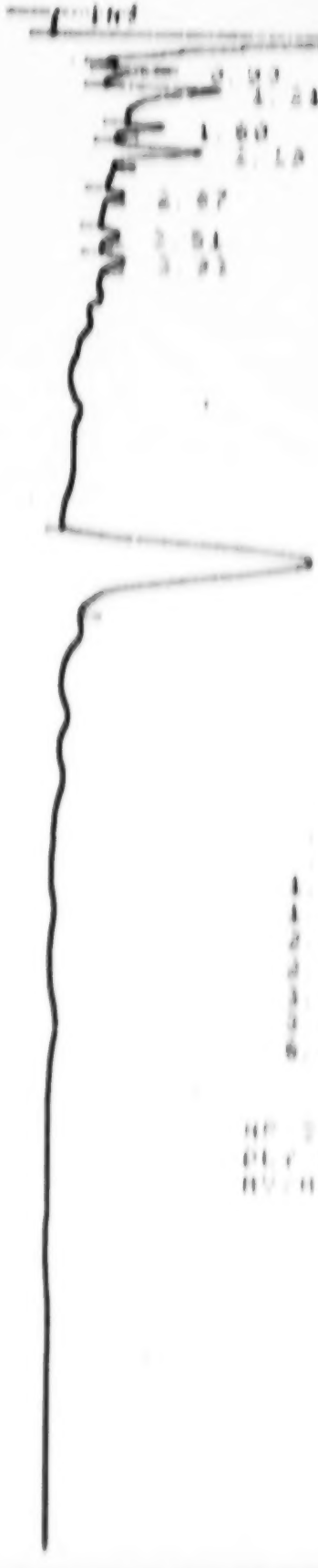


Figure B-10. Chromatogram of Typical Borosilicate Water Sample.

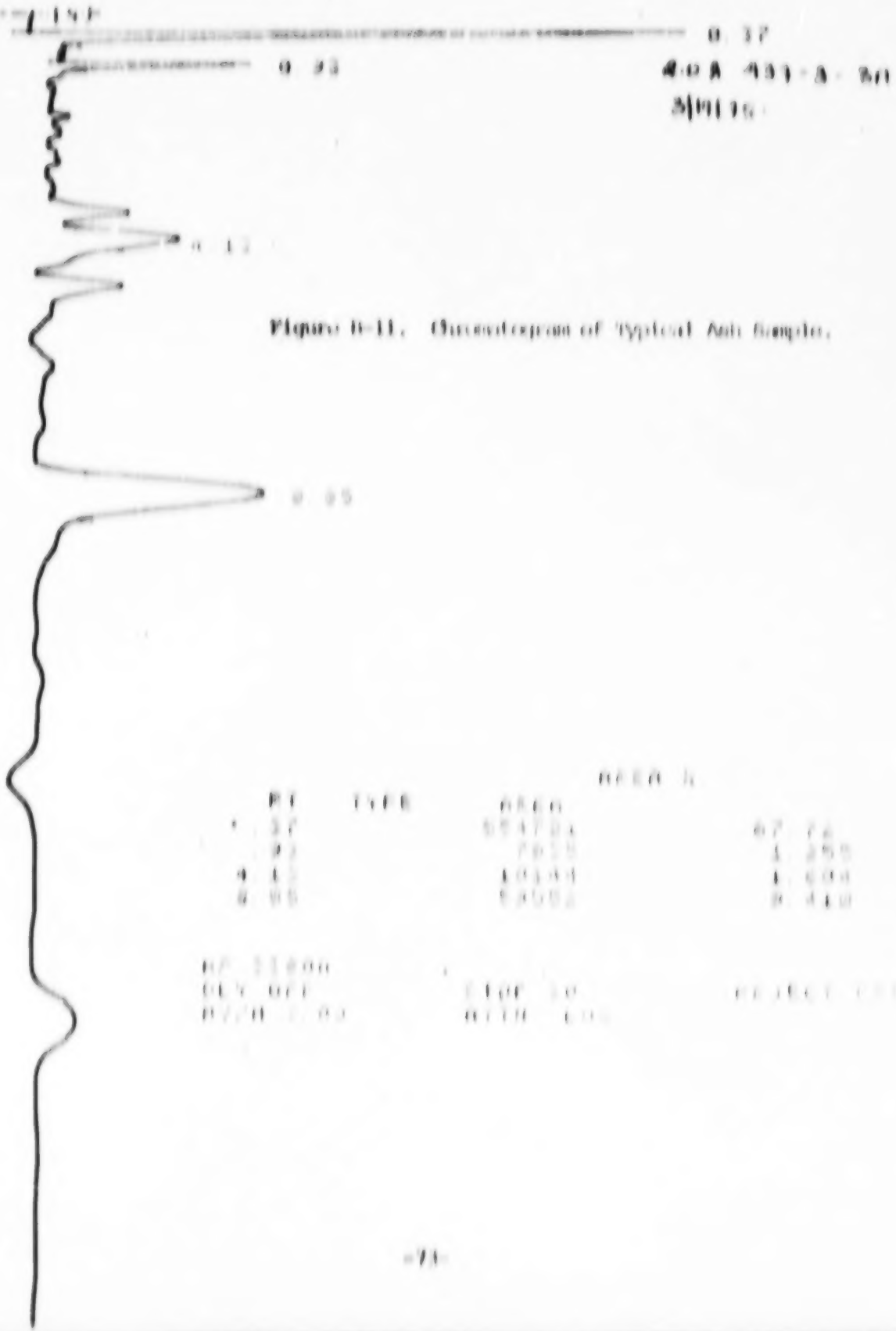


Figure B-11. Chromatogram of Typical Ash Sample.